GE-MTSD-R-059

PYROTECHNIC HAZARDS CLASSIFICATION AND EVALUATION PROGRAM

PHASE III SEGMENTS 1 - 4

INVESTIGATION OF SENSITIVITY TEST METHODS AND PROCEDURES FOR PYROTECHNIC HAZARDS EVALUATION AND CLASSIFICATION

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PART A

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FOREWORD

The studies described in this report comprise Phase III, Segments 1 through 4, of Edgewood Arsenal's three-phase Pyrotechnics Hazards Evaluation and Classification Program. The report was prepared by the General Electric Company, Management and Technical Services Department (GE-MTSD), Bay Saint Louis, Mississippi, under National Aeronautics and Space Administration (NASA) Contract NAS8-23524 for the Engineering Test and Evaluation Section, Process Technology Branch, Chemical Process Laboratory, Weapons Development and Engineering Laboratory, Edgewood Arsenal, Edgewood, Maryland.

The work described herein was performed in accordance with the contract workscope with technical direction and assistance from W. P. Henderson, Chief, Engineering Test and Evaluation Section. Mr. A. E. Becker and Mr. J. Vogelein of the Edgewood Safety Office, in conjunction with Mr. Henderson, were instrumental in structuring the total pyrotechnics hazards program, which was comprised of the following phases and segments (For reference purposes the appropriate GE-MTSD report number has been indicated):

- Phase I (GE-MTSD R-035) was comprised of two segments. Segment 1 encompassed TB-700-2 testing of a number of pyrotechnic compositions and end items, and Segment 2 covered "TNT equivalency" testing of these same compositions in granular form.
- Phase II, the study of hazards associated with pyrotechnic manufacturing processes, consisted of seven segments as follows:
  - Segment 1 (GE-MTSD R-045) reported on the findings of a comprehensive records and experience analysis of accidents and incidents throughout the pyrotechnic industry.
  - Segment 2 (GE-MTSD R-040) contained the findings and recommendations arising from an operational survey of Pine Bluff Arsenal.
  - Segment 3 (GE-MTSD R-054) was the test plan developed for the increments of work, Segments 4, 5, 6, and 7.
  - Segments 4, 5, 6, and 7 (GE-MTSD R-058) included the test description and results of all Phase II tests. Conclusions and recommendations based on the test data were applied to new techniques and concepts of process hazard minimization.
- Phase III which is reported in this volume includes the results of investigations into the properties of pyrotechnic compositions and the methods by which they might be more reliably and precisely evaluated and classified.

Related studies reported on previously and technically administered by the Engineering Test and Evaluation Section were as follows:

- Effects of Copper and Heavy Metals on the Sensitivity of Pyrotechnic Mixes (GE-MTSD R-036)
- XM-9 C/S Canister End Item Tests (GE-MTSD R-037)
- Electrostatic Vulnerability of E-8 and XM-15/XM-165 Clusters - Phases I & II (GE-MTSD R-052 and R-057)

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Edgewood Arsenal technical personnel who contributed significantly to the success of this program are as follows:

- W. P. Henderson, Supervisor, Engineering Test and Evaluation Section
- W. P. Junkin, Chief, Chemical Process Laboratory
- J. K. Bartell, Chief, Process Technology Branch
- A. E. Becher, Chief, Safety
- J. Vogelein, Safety Engineer

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The following General Electric Materiel Testing and Research Project engineers were responsible for the successful completion of their respective tasks:

- Section 2 - Segment 1 - J. Pankow
- Section 3 - Segment 2 - J. Pankow
- Section 4 - Segment 3 - J. Pankow
- Section 5 - Segment 4 - J. Pankow and A. Lasseigne

Additional program support was provided as follows:

- Data Acquisition - S. Russell
- Data Analysis - W. Stone
- Technical Support - T. Small and P. Fassnacht

All of the projects were under the technical direction of P. V. King, Sr., Manager of the Materiel Testing and Research Group, and D. M. Koger, KEMOPS Program Manager.

Special mention must be made of the competence and efficiency of the entire test team, led by Mr. F. L. McIntyre, Test Supervisor, and S. Fuentes, Lead Technician and Ordnance Specialist, who worked diligently in the face of many obstacles to provide the valid, meaningful data in the minimum time.

To list the many members of the GE-MTSD team at the Mississippi Test Facility that supported the program in a commendable manner would not be practical in this report. However, to many of these dedicated personnel, another grateful thanks.

ABSTRACT

This report contains the findings, conclusions, and recommendations relative to the investigations conducted to evaluate current tests, propose modifications, and propose additional tests to classify pyrotechnic materials and end items as to their hazard potential. In the past, pyrotechnic compounds were subjected to the same classification test series as were high explosives (as specified by TB 700-2), even though their reaction characteristics are quite different. This situation has prevailed because of a lack of information to determine applicable tests and a lack of data to establish the tests' validity; thus, the study reported herein is intended to provide information required to establish an applicable means of determining a pyrotechnic's hazard potential.

The existence of (or degree of) a hazard potential of a reactable system may be defined in terms of its probability of progressing through each of the ICT elements (initiation, communication, and transition to detonation). From a safety standpoint, it is desirable to minimize the initiation probability, but in most cases the criteria affecting the safety regulations of reactable system configurations are established independently of the initiation probability. Thus, it is primarily the communication and transition aspects of the systems which require determination and classification. For example, when functioning as designed, the transition probability of pyrotechnic items approaches zero, but under confinement a reaction transition or an equivalent effect may be quite likely. A measure of the energy released during this process is the relative percentage of a standard high explosive (HE) detonated at the same location which would produce an identical effect. The effect traditionally chosen is that of the peak overpressure or impulse of the resulting shock wave as a function of distance from the source. In order to better understand this critical characteristic of pyrotechnics, HE equivalency testing and evaluation comprise a major portion of this report. Other hazard classification tests investigated and evaluated include dust ignition sensitivity and combustibility, instrumented impact ignition sensitivity, spark ignition sensitivity, differential thermal analysis, and Parr bomb.

EXECUTIVE SUMMARY

DESCRIPTION OF PROGRAM PHASES

This report deals with findings and recommendations of the third and final portion of Edgewood Arsenal's preliminary Hazards Evaluation Program, which was begun March 17, 1969 to provide the foundation for cost effective solutions to operational and safety problems associated with current production facilities, and to provide an approach to the many problems in the overall Arsenal modernization program.

As outlined previously, this program was structured into three phases to provide a definable technological base upon which to build a completely new family of safety criteria applicable specifically to pyrotechnics.

PHASE I - HAZARDS CLASSIFICATION TESTS

Phase one dealt with:

a. The examination of the properties and hazard classification of a number of standard pyrotechnic munitions and their ingredients in accordance with currently acceptable criteria.

b. Examination and discussion of the appropriateness of such criteria.

c. The determination by several test methods, of the so called "TNT equivalency," of the munitions and ingredients in question.

d. Recommendations for further and more definitive testing in Phase 3.

PHASE II - OPERATIONAL HAZARDS ANALYSIS/DEVELOPMENT AND PROTECTIVE CONCEPTS AND CRITERIA

Phase II included:

a. An operational hazards analysis of a governmental production facility and analysis of several operations of contractor facilities.

b. A systems analysis to evaluate "maximum credible" and "worst case" incidents and to develop appropriate scaled test simulations. Included in each evaluation were all identified contributors to potentially synergistic reactions.

c. Conduct of tests at various scale sizes to simulate worst case conditions.

d. The development of facility construction and operational shielding criteria and concepts.

e. The proof testing of such criteria/concepts. One concept developed appears to represent a breakthrough in the state-of-the-art.

PHASE III - DEVELOPMENT/MODIFICATION OF HAZARDS CLASSIFICATION CRITERIA AND TEST METHODS

In this, the last phase of the Hazards Evaluation Program, an attempt has been made to identify the fundamentals of a proposed new method of classifying pyrotechnics, (and hazardous materials).

This includes the following:

a. Identification of information needed to satisfy the proposed new classification criteria.

b. Identification of currently accepted tests which provide some of this information.

c. Recommended modifications to current test/classification devices.

d. Recommendations for test to develop new methods to fit the proposed classification criteria.

The remainder of this summary will discuss the Phase III activities.

BACKGROUND

Safety authorities generally (including the ASESB, and the AMC, and Edgewood Arsenal Safety Offices) have long recognized that existing hazards classification criteria, which have resulted from a large body of historical, and empirical data furnished by serious accidents, and supported by R&D Programs conducted to supply information applicable to high explosives and propellants, were not suitable for pyrotechnics operations. Often overlooked was the fact that adherence to these criteria, or to criteria developed by analogy not only failed to assure the optimum degree of safety (since it tended to provide protection against the wrong hazards) but also represented a significant cost consideration in the design and operation of pyrotechnics facilities, and in the transportation of such munitions.

The advent of the Edgewood Arsenal modernization program made it imperative that these factors be adequately considered if the goals of increased production and safety, at reduced cost, were to be obtained.

In the effort reported on herein, an attempt has been made to structure a new hazard evaluation concept, namely that hazardous materials should be classified and ranked by their ability to initiate, communicate (and upgrade initiation) and to transit to detonation when subjected to the various stimuli available in the environment. This concept referred to herein as the ICT criteria has been discussed with safety authorities at various government levels. Ideally, materials thus classified would be rated as to the probability of any or all events occurring when the materials are subjected to the various environmental stimuli.

Classification by these criteria will in turn serve to indicate more accurately the nature of the hazards and the type of protection required in a given situation. This concept is applicable to the operation of "Hazardous" or Dangerous" materials from oxidizers and flammables through high explosives.

While a complete resolution of the problem was beyond the scope of the current investigation, sufficient information has been gained to permit modification of currently used test and classification equipment to furnish some of the information required. Other existing tests which are appropriate have been identified and recommendations for further modification and new test development have been included herein.

DISCUSSION OF EXISTING TEST METHODS

The great majority of tests currently used by the explosives/propellants/pyrotechnics industry are aimed at indicating in a general way the materials' sensitivity to ignition by various stimuli, (I as previously discussed).

Several indirectly indicate communicability and upgrading (C) and several tend to indicate detonability (T). As discussed Phase I report, almost all these methods are highly suspect when applied to the pyrotechnics tested in this contract and only one test which is extremely "operator sensitive" (Impact Test) resulted in the Class 7 classification of a few of the materials tested in Phase I (see GE-MTSD Report No. 035).

Several improvements to this test apparatus proposed by the contractor, including one developed in a series of tests conducted for Picatinny Arsenal, Contract No. NAS8-25149, appear to have merit and should be considered.

Ignition and unconfined burning tests (described in Phase I and TB 700-2) tend to provide information useful for calculation of communication hazards (C) but are inexact and provide only for observation of an explosion of an adjacent cube of material but no measurement of the severity of the explosion.

Similarly, end item tests which give data useful to the evaluation and classification of a given pyrotechnic in a given package represent a solution for the specific combination only, and tests reported on herein have shown that classification by analogy may be dangerous.

In a similar way, most of the tests included in TB 700-2 are not really applicable to pyrotechnics since they represent an attempt to classify items that burn by techniques designed to indicate a detonation.

Modifications proposed herein will increase the usefulness of a number of these tests by providing data more directly applicable to the problem, and by providing for accumulation of quantitative rather than qualitative data. Other tests which are important in evaluating the hazards potential such as the Hartmann, DTA, and others are also discussed herein.

TNT (HE) Equivalency

Much attention has been devoted in recent years to the concept of TNT equivalency which means literally that the material in question is compared to TNT (or usually spherical pentolite) in terms of the ratio of pressures generated by given weights of HE or pyrotechnics at a given distance, or by the ratio of weights required to given identical pressures at these distances.

In the evaluations and tests reported on herein, we have discussed the various methods of confining TNT equivalencies. Insofar as pyrotechnics processors are concerned, it is probably simpler to use the equivalent weight concept; i.e., the equivalent weight of HE required to generate the overpressure of concern at a given point; eg. a 10 percent equivalency value would mean that 100 lbs. of material reacting would produce at a given distance the same peak overpressure as 10 lbs. of TNT at the same distance. In these terms, none of the pyrotechnic smoke compositions tested resulted in "equivalencies" greater than 15 percent. All, in fact, were within the range of pressures that could be attained by a pneumatic rupture of a pressure vessel of the volume and wall thickness as the test vessel.

All indications to date are that if a detonation of any of the pyrotechnics tested is possible, it would require a donor charge not available in its normal environment or a degree of confinement many times greater than that attainable by accident.

Another misleading factor arises from the more or less standard practice of evaluating materials exploded in containers (or otherwise confined) with a reference curve developed for bare high explosives which introduces a further error since a high explosive which is confined has a greatly reduced HE equivalency.

Of more importance to Pyrotechnics Hazards Evaluation problems is the fact that the rate of pressure rise of high explosives initiated in a confining vessel is measured in fractions of milliseconds, whereas the pressure rise for pyrotechnics is measured in tens of milliseconds. Amont other things, this indicates that venting, suppressing and attenuating techniques will be more effective in pyrotechnics application than in high explosives applications.

Summation:

- An effective beginning has been made in developing new criteria for classification of hazardous materials.
- Appropriate modifications to existing test methods to increase their effectiveness for application to pyrotechnics have been developed.
- Additional tests (not previously listed in TB 700-2) have been evaluated and suggestions made for their application and modification as appropriate.
- The concept of explosive equivalency has been explored in detail with appropriate recommendations.

- Considerable information regarding the nature of the reactions of pyrotechnic smoke mixtures to various stimuli has been attained and correlated.
- Recommendations for modification of current classification criteria have been developed.

Conclusion:

The result of all findings to date is that the objectives originally outlined, namely increased safety and reduced cost are attainable, and that the appropriate modification of Hazards Classification criteria will help greatly in meeting these objectives.

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SECTION 1
INTRODUCTION

1.1 GENERAL

This document constitutes the final report of a study of a classification system appropriate for the establishment of the hazard potential of pyrotechnic compounds and items. This study comprises Phase III of a comprehensive three-phase Hazards Evaluation Program (HEP) being conducted by the General Electric Company, Management and Technical Services Department (GE-MTSD), Bay Saint Louis, Mississippi, under National Aeronautics and Space Administration (NASA) Contract NAS8-23524 for the Engineering Test and Evaluation Section, Process Technology Branch, Chemical Process Laboratory, Weapons Development and Engineering Laboratory, Edgewood Arsenal, Edgewood, Maryland.

Phase I of the HEP consisted of applying the existing hazards classification techniques (as defined in TB 700-2) to pyrotechnic items of concern. Phase II comprised a study of the hazards associated with the manufacture of pyrotechnic items. Thus, this Phase III study utilized a wealth of data, experience, and knowledge from previous investigations (additional relevant programs are discussed in the Foreword).

1.2 STUDY ORGANIZATION

Phase III was contractually and functionally divided into four segments. The contents of the four segments are defined as follows:

- Segment 1 (reported in Section 2 of this report) - Define the performance characteristics required to evaluate pyrotechnics and evaluate existing explosive classification test methods for application to pyrotechnics classification and performance.
- Segment 2 (Section 3) - Develop data required for the modification of existing standards.
- Segment 3 (Section 4) - Develop new and/or modify existing equipment and test methods to obtain required data and conduct validation tests as appropriate.
- Segment 4 (Section 5) - Prepare and submit data requested for testing, evaluation, and classification of pyrotechnic materials.

The results of these incremental studies are reported herein in the sections indicated. Contract compliance may be verified by comparison with the content of the appropriate sections. A logic diagram of the Phase III effort is shown in Figure 1-1.

SECTION 2
SEGMENT 1 - DEFINITION OF PERFORMANCE CHARACTERISTICS REQUIRED TO EVALUATE PYROTECHNICS AND EXISTING EXPLOSIVE TEST METHODS

2.1 BACKGROUND

The need for an appropriate hazards classification is obvious when one considers the quantity of reactionable compositions and items which are transported, handled, and stored. The great variety of materials and their subsequent variations in reaction characteristics making some inherently more hazardous than others requires an appropriate classification structure and testing criteria to establish the material's position within the classification scheme. Appropriate and properly performed classification criteria can then be utilized to establish safe transportation, handling, and storage regulations for all materials of concern.

2.2 CURRENT PYROTECHNIC HAZARDS CLASSIFICATION CRITERIA

The existing hazard classification standards are contained in manual TB 700-2, "Explosives Hazard Classification Procedures." The purpose of TB 700-2 is to set forth procedures for determining the reaction of ammunition, explosives, and solid propellants to specified initiating influences. Based on reactions obtained, it further provides for assignment of appropriate hazard classifications (Quantity-Distance Class, Storage Compatibility Group, DOT Class and DOT Markings). Cognizant safety authorities have questioned the validity of subsequent hazard classifications of pyrotechnics based on the tests and techniques required in TB 700-2. It is felt that the tests are most applicable to high explosives materials classification and leave serious deficiencies when applied to pyrotechnics.

The classification tests are described in Chapters 3 - 5 as follows:

- Chapter 3 - Minimum Test Criteria for Bulk Explosive Compositions and Solid Propellant Compositions.

  "Tests in this chapter are intended to develop data on the stability and sensitivity of new compositions of bulk explosives and solid propellants. Such data is required in order to determine that these compositions are safe to handle, transport, and store."

- Chapter 4 - Minimum Test Criteria for Ammunition and Explosive Items Including . . . Pyrotechnics, . . .

  "The tests in this chapter are intended to develop data upon which storage and transportation classifications of ammunition items may be based."

Table 6 in Chapter 4 contains the "Minimum Test Criteria for Determining Hazard Classification of Pyrotechnics" and is included in Table 2-1 (this report) for reference. Note that these tests are simulations of conditions expected to occur during typical accidents.

- Chapter 5 - Minimum Test Criteria for Rocket Motors or Devices Containing Solid Propellants. The tests outlined in this chapter are not applicable to pyrotechnics hazard classification.

TB 700-2 test criteria are not intended to determine the "hazards during various stages of manufacture and assembly" (the subject of the Phase II study, GE-MTSD-R-045, 040, 054, and 058) on the "susceptibility to accidental initiation by electrostatic and electromagnetic influence" (the subject of the electrostatic vulnerability studies of E8 and XM15/XM165 clusters, GE-MTSD-R-052 and 057).

The classification criteria required in TB 700-2 are primarily based on simulation of likely accidental environments. It fails to establish a test methodology consistent with a thorough, appropriate analysis of the conditions required to result in a hazardous situation. An inherent sequence of events must occur in any accident involving reactable materials. This cause and effect approach to the problem is formalized in the following paragraphs.

The classification procedure as defined by TB 700-2 rates bulk materials in the following categories:

- DOT Forbidden - If spontaneous ignition is possible within the temperature environment range anticipated during transportation and storage.
- DOT Restricted - If ignition is possible because of impacts anticipated during normal handling and transportation.
- DOT Class A, Military Class 7 - If an external detonation can be easily induced in and propagated through the material or ignition is possible because of impacts encountered during abnormal handling.
- DOT Class B, Military Class 2 - If material is reactive but ignition is not likely except when subjected to extreme accidental conditions and even then the material's probability of detonating is minimal.

The Chapter 3 (TB 700-2) tests are used to establish the composition's position within the above hazard classification levels. The classification interpretation specified for the test results is shown in Figure 2-1. Note the objective of this procedure is to establish a classification scheme which can be conveniently related to the condition encountered during transportation, handling, and storage.

Table 2-1. TB 700-2, Chapter 4, Table 6. Minimum Test Criteria for Determining Hazard Classification of Pyrotechnics - All Types and Certain Small Items Containing Solid Propellants

1. Type	2. Packaging, as Normally Stored and Shipped	3. Type of Info to be Determined by Test	4. Types of Initiation to Obtain Info Outlined in Item 3
Burning	Individual Item or Unit	Propagation Within a Single Container	Simple Ignition
Detonating	More Than 1 Item Per Unit	Propagation from 1 Container to Another	Detonation
		Determination of Fragment Hazard	External Heat
		Determination of Blast Hazard	
		Determination of Fire Dispersement Hazard	

5. Minimum Test Criteria

Type Test	Number Items Per Test	Number of Tests	Priming	Booster	Confinement
Test A. Detonation	1 Container	5	Normal Means of Ignition or Engr Special Blasting Cap	None	None
Test B. Detonation	2 Containers	5	Same as Above	None	None
Test C. External Heat	1 to 6 Containers Depending on Size of Unit	1	None	None	Steel Banded

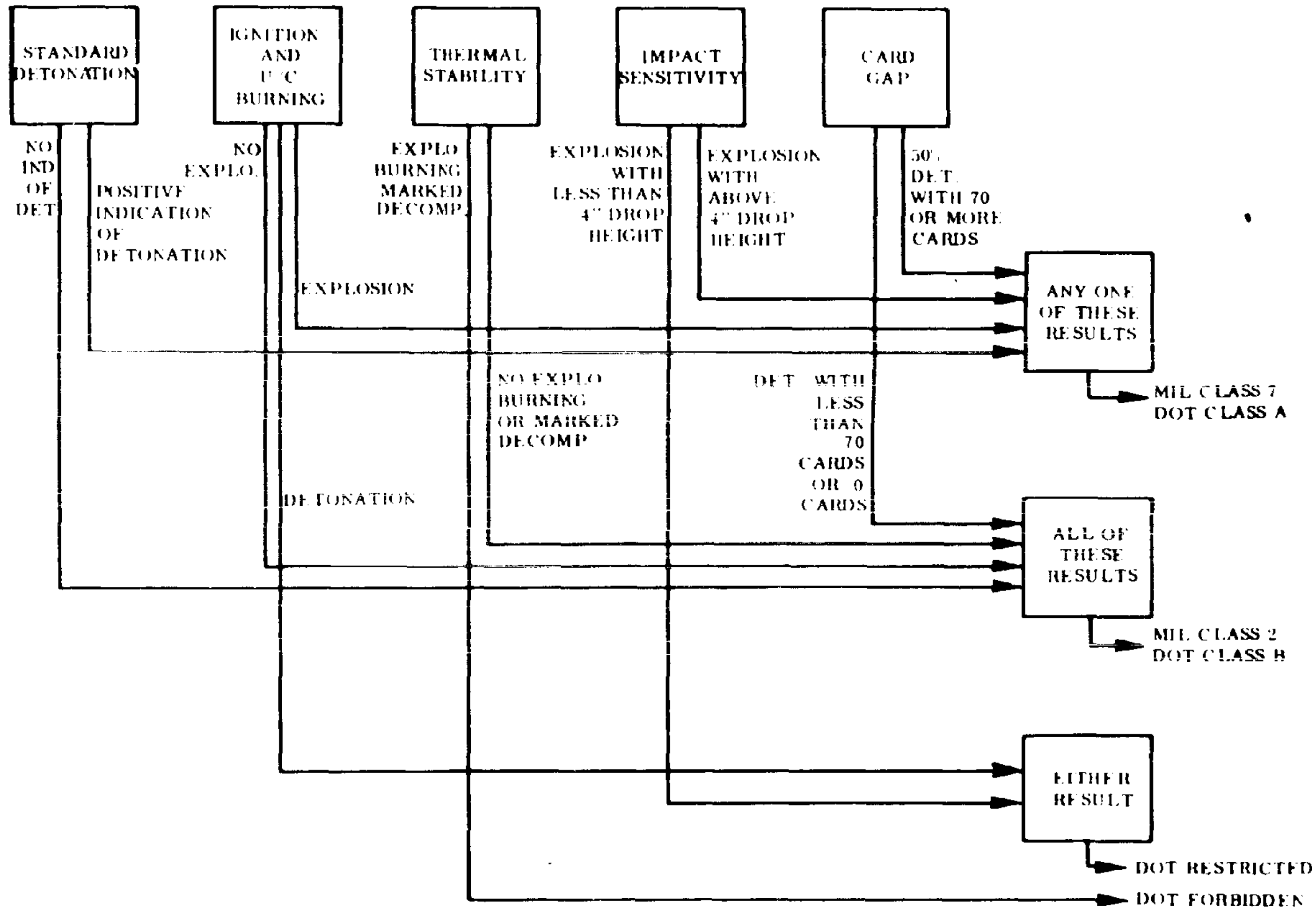

Figure 2-1. Relationship of TB 700-2 to Classification Requirements

2.3 THE ICT APPROACH

2.3.1 GENERAL

The following paragraphs present, with amplifications, the following physical/chemical phenomena, some or all of which normally occur sequentially during the reaction development from initiation to detonation when a pyrotechnic mixture (solids and dusts) is ignited:

- Initiation at a localized region.
- Communication to adjacent material by a subsonic burning process (deflagration).
- Transition from deflagration to detonation.
- Propagation of detonation.

This prerequisite sequence of events is referred to as the ICT series. The following discussion is intended to briefly summarize the results of recent investigations as it relates to ICT rather than to fully describe the kinetics of chemical reactions of pyrotechnic mixes.

2.3.2 INITIATION CONSIDERATIONS

That explosions are thermal in origin is widely accepted. According to the "hot spot theory," energy must be transformed into heat to give a "hot spot" of suitable size and temperature to support growth. At a microscopic scale, ignition of a reactable granular material is caused by:

- Adiabatic compression of trapped air pockets
- Intergranular friction
- Granular-container wall and intragranular friction
- Heat injection

Any of these mechanisms is capable of generating a "hot spot" inducing a chemical reaction. If the reaction is exothermic and the energy in the "hot spot" is above a critical threshold level, the reaction will be self-sustaining, thus initiating the material.

One or more of the microscopic ignition mechanisms may be stimulated by the mechanical/electrical effects induced during manufacturing processes. These microscopic initiating mechanisms include:

- Pressure
- Friction
- Heat transfer
- Electrostatic discharge

The magnitude of the contributions of these effects is dependent upon the manufacturing process involved and whether it is operating under normal or abnormal conditions.

2.3.3 COMMUNICATION CONSIDERATIONS

The reaction front communicates with the adjacent unreacted material propagating the reaction. In some materials, an increase in reaction rate accompanies the propagation of the reaction front. For a significant increase of the reaction rate to occur, the heat generated by the chemical reaction must be liberated at a greater rate than is necessary to sustain the combustion and to balance the conductive heat losses.

From experimental investigations, the following factors have been identified as influencing the burning rate of pyrotechnics:

- Degree of confinement
- Surrounding gas pressure
- Density
- Temperature
- Cross-sectional area of combustion zone
- Nature of chemical reaction process
- Rate of heat loss

Once initiation has occurred, the reacting material communicates with the adjacent unreacted material providing a mechanism for propagation of the reaction front. The reaction may be such that the reaction front velocity is accelerated until it exceeds the velocity of sound in the host material. This occurrence is referred to as a transition from deflagration to detonation.

The reaction front velocity in a material is a function of the material's reaction rate--its chemical properties, its dispersal, pressures, and temperatures. In general, the more exothermic the reaction and the larger the exposed surface-to-volume ratio, the higher the rate of reaction. Likewise, the smaller the difference between the initiation temperature and the ambient temperature, the easier it is for the reaction to propagate. High ambient temperature and pressure reduce this difference.

All parameters except the chemical properties are considerably affected by the degree of confinement of the material. The classes of confinement configurations include:

- Unconfined Material
- Vessel Confinement - Pyrotechnic material in a closed, rigid chamber. This investigation includes determining whether the blast overpressure released is a characteristic of the deflagrating/detonating pyrotechnic or is a result of pneumatic rupture of the vessel caused by buildup of pressure during burning.
- Self Confinement - Large mass alone as its own confining medium.

The pyrotechnic material may be dispersed as a dust suspended in air. The communication properties of this distribution may readily lead to a transition to detonation. Variations in dust density may be required to induce a transition. Such a condition is referred to as inducing a runup reaction. The terminal element in a runup chain may be a concentration of pyrotechnic powder which, if able to maintain the detonation through its bulk, would result in a violent explosion.

2.3.4 TRANSITION FROM DEFLAGRATION TO DETONATION

A number of investigators have noted that the culminative action of a deflagration process, whose reaction front propagates with increasing pressure and temperature, is its transformation into a shock wave. It is during transition that the reaction front transforms from a subsonic to a supersonic wave. Thus the reaction undergoes a transition from deflagration (burning) to detonation, forming a shock wave reaction front.

It has not been determined, experimentally, whether the mechanisms required to transform deflagration into detonation are the same for pyrotechnics and explosives.

2.3.5 DETONATION PROPAGATION

In a general sense, the ability of a solid to maintain a reacting compression wave is referred to as its ability to support propagation. High speed photographic studies by several investigators on thin films of azides and fulminates have shown that the following processes contribute to propagation of detonation:

- Creation of a dust-like atmosphere by the action of the shock front breaking up the solid into fine particles, thereby increasing the material's surface-to-volume ratio and, consequently, the reaction rate.
- Shock initiation whereby gas pockets ahead of the reaction zone are compressed and serve as ignition sources to maintain the shock wave. In some materials, intercrystalline friction can also result in providing hot spots ahead of the reaction zone.

A thorough literature survey has served to reemphasize the fact that the events leading from initiation to detonation are not completely understood, and that at least one new theory has been developed recently. These findings, together with test results, have indicated a need to conduct additional studies, particularly in the area of pyrotechnic reactions.

2.3.6 HUMAN VULNERABILITY FACTORS

Although it is not an objective of the current program to express results in terms of damage to a human body, a discussion of body vulnerability is included to facilitate better understanding of the rationale for this study as it ultimately relates to the vulnerability of the human body and the applicability of the data.

There are three primary categories of hazards to the human body associated with an explosion; namely, blast, thermal, and fragmentation. They are defined as follows:

a. Blast - Blast injuries are classified as being either direct or indirect. White and Richmond (reference Appendix H, Doc. 250) have reported that three parameters of the blast wave affect the extent of the direct injuries to the body: (1) the rate of pressure rise at the blast wave front, (2) the peak overpressure attained, and (3) the duration of the positive phase of the overpressure. Indirect blast injuries are associated with the impact of missiles, either penetrating or non-penetrating, and the physical displacement of the body as a whole.

b. Thermal - Thermal injuries may result through either radiation or direct contact with pyrotechnics being sprayed or dispersed.

c. Fragmentation - Fragmentation injuries are possible if high velocity fragments result. The relationship between fragment mass, velocity, and density that will cause injury upon impact with the body is indeterminate. It has been generally concluded that any wound causing a serious cavity in the body can be considered lethal. The threshold for such an injury can be taken to be almost 100 feet per second for a 10-gram fragment. For smaller fragments, the threshold velocity is, of course, higher.

An interesting result of our studies is that blast overpressure resulting from incidents involving pyrotechnics is not likely to be of sufficient magnitude to provide a significant hazard to the human body.

2.4 APPLICATION OF ICT TO HAZARDS CLASSIFICATION

The ICT formalism not only provides a realistic and meaningful analysis of the principles involved in reaction growth, but it also establishes an appropriate structure on which a more realistic approach to hazards evaluation can be developed. The existing TB 700-2 tests applicable to pyrotechnics are listed in Table 2-2, and the applicable element of the ICT sequence which is measured by the test is indicated.

Since the objective of TB 700-2 is limited to establishment of the hazard classifications appropriate to transportation, handling, and storage, the methods of initiation considered are restricted to those encountered during logistical procedures. These include thermal ignition such as would be encountered by prolonged exposure to direct sunlight or within a container exposed to solar heat and impact induced ignition as occurs when the containers are jostled by vehicle accelerations and decelerations and irregularities in the road surface, or due to normal handling, such as dropping into position, shoving into place, etc. In addition to these normal stimuli, there are those as a result of an inadvertent accident, such as a fire or detonation of adjacent material. All of these mechanisms are appropriately simulated by existing TB 700-2 tests.

Table 2-2. Correlation Between Candidate Tests and ICT

		Par.	Candidate Test	Initiation	Communication	Transition
Existing TB 700-2 Tests	Bulk Materials	3-8	Detonation			x
		3-9	Ignition & Unconfined Burning	x	x	
		3-10	Thermal Stability	x		
		3-11	Impact Sensitivity	x		
		3-12	Card Gap	x	x	x
	Shipping Configuration	Type				
		A	Single Shipping Container, normal ignition		x	x
		B	Two shipping containers, normal ignition		x	x
		C	Shipping containers subjected to external heat	x	x	x
Proposed Tests			Confined HE Equivalency			x
			Differential Thermal Analysis	x		
			Parr Bomb		x	x
			Instrumented Impact Sensitivity *	x		
			Dust Ignition Sensitivity **	x		
			Spark Ignition Sensitivity **	x		

* Instrumented version of original impact sensitivity test

** External to scope of current TB 700-2 objective

The primary communications problems involved are inter and intra container. These tests are also included in current TB 700-2 testing.

Determination of the capability of transition to detonation is considered in all simulation tests. The bulk material unconfined burning test and detonation test are performed to attempt to induce detonation under conditions of minimal confinement. Normal vessel confinement effects are recorded during the tests of filled shipping containers. In addition, the capability of an adjacent detonation to induce a detonation in the sample material is established via the card gap test.

The conclusion must therefore be reached that current TB 700-2 tests do simulate appropriate conditions to adequately determine the ICT characteristics of a potential accident involving pyrotechnics when limited to the conditions present during transportation, handling, and storage (excluding manufacturing/processing environments and electrostatic ignition, as specified in TB 700-2). Some modifications of these tests are appropriate in order to obtain data which is more quantitative in nature, and a thorough analysis of the interpretation techniques to provide a meaningful hazard classification is recommended. In addition, other tests to extend the applicability of classification data to manufacturing hazards and situations involving electrostatic ignition are suggested. Some of the tests proposed to extend classification to the more stringent conditions encountered during manufacturing were evaluated in Segment 3 (Section 4 of this report) and are included in Table 2-2.

SECTION 3
SEGMENT 2 - DEVELOP DATA REQUIRED FOR THE RECOMMENDED MODIFICATION OF EXISTING STANDARDS

3.1 INTRODUCTION

In this segment, the tasks outlined in the contract workscope were as follows:

a. Perform laboratory and field tests with existing techniques and equipment to obtain required data such as heat of reaction, friction sensitivity, electrostatic sensitivity, shock sensitivity, and thermal sensitivity.

b. Identify inadequacies of the current test equipment and techniques.

Tests performed under (a) above were designed to provide information that was found to be lacking as a result of the Phase I test program. The test series discussed in this section are instrumented impact sensitivity, Parr Bomb, Differential Thermal Analysis, and Electrostatics. A discussion of some of the anomalies in the TB 700-2 tests found during Phase I is also included in this section and serves to satisfy in part the requirement of (b) above.

3.2 LABORATORY AND FIELD TESTS

3.2.1 DIFFERENTIAL THERMAL ANALYSIS

Differential thermal analysis (DTA) is used to determine physical and chemical reactions that might occur when the sample material is subjected to a rise in temperature.

DTA measurements are used extensively to detect any exothermic or endothermic changes that might occur in a chemical system by measuring the temperature difference between a sample and a thermally inert reference material as both are heated at a constant rate of increase of temperature. The reference material selected should not undergo any thermal reaction over the temperature range under investigation, so that any exothermic or endothermic change occurring within the sample will cause its temperature to either exceed (exothermic) or lag behind (endothermic) that of the reference material during the course of a physical or chemical reaction.

All physical or chemical reactions that occur during an analysis are related to the mass of the sample, the size of the sample, the heating rate of the sample, and the particle size of the sample. These chemical or physical reactions represent changes that may be related to initiation, decomposition, dehydration, crystalline transition, melting, boiling, vaporization, polymerization, oxidation, and reduction of the material under investigation.

The process of ignition involves heating the material to its ignition temperature which is the minimum temperature required for the initiation of a self-sustaining reaction. An ignition stimulus, which can be reduced to the effect of heat absorption, starts a sequence of pre-ignition reactions involving crystalline transitions, phase changes or thermal decomposition of one or more of the ingredients. The DTA ignition temperature values are listed in Table 3-1.

Table 3-1. Differential Thermal Analysis Values for Selected Pyrotechnic Compositions

SAMPLE	IDENTIFICATION	IGNITION TEMPERATURE °C
$KClO_3$ - Sulfur		179
HC White Smoke	HC	193
Fuel Mix	FM 3-69-1	193
Sulfur Green	SG 3-69-1	196
Sulfur Yellow	SY 3-69-1	196
Lactose Red	LR 3-69-1	197
Sulfur Red	SR 3-69-1	201
Lactose Violet	LV 3-69-1	210
Lactose Yellow	LY 3-69-1	217
Sulfur Violet	SV 3-69-1	221
Lactose Green	LG 3-69-1	332

The data is shown in order of increasing sensitivity to ignition with HC white smoke composition the most sensitive composition (lowest ignition temperature) and lactose green the least sensitive. These values along with the other empirical data taken from other Phase I and Phase III tests will be compared in an effort to correlate the various ranges of sensitivity values. An examination of the DTA data by itself indicates all of the compositions are in a fairly tight range of values (193 - 221°C) with the exception of lactose green. There is no readily plausible explanation for the relatively high value for lactose green except that it might be due to sampling error.

It should be noted that the DTA value for pure potassium chlorate - sulfur (stoichiometric mixture) is most sensitive. This sensitivity for $KClO_3$ -S mixtures is borne out in all of the other Phase I and Phase III tests and is, of course, basis for the recommendation made now and many times previously; i.e., that $KClO_3$ and sulfur compounds must be kept separated until such time as they are desensitized by one or more of the other additives.

All DTA data in this report were obtained on a Fischer Series 200 differential thermal analyzer (Figure 3-1). Thermograms were produced by a Varian Aerograph Model 20, dual-channel, strip-chart potentiometric recorder having a 1 MV full-scale sensitivity on each channel. A typical recorder trace (thermogram) is shown in Figure 3-2.

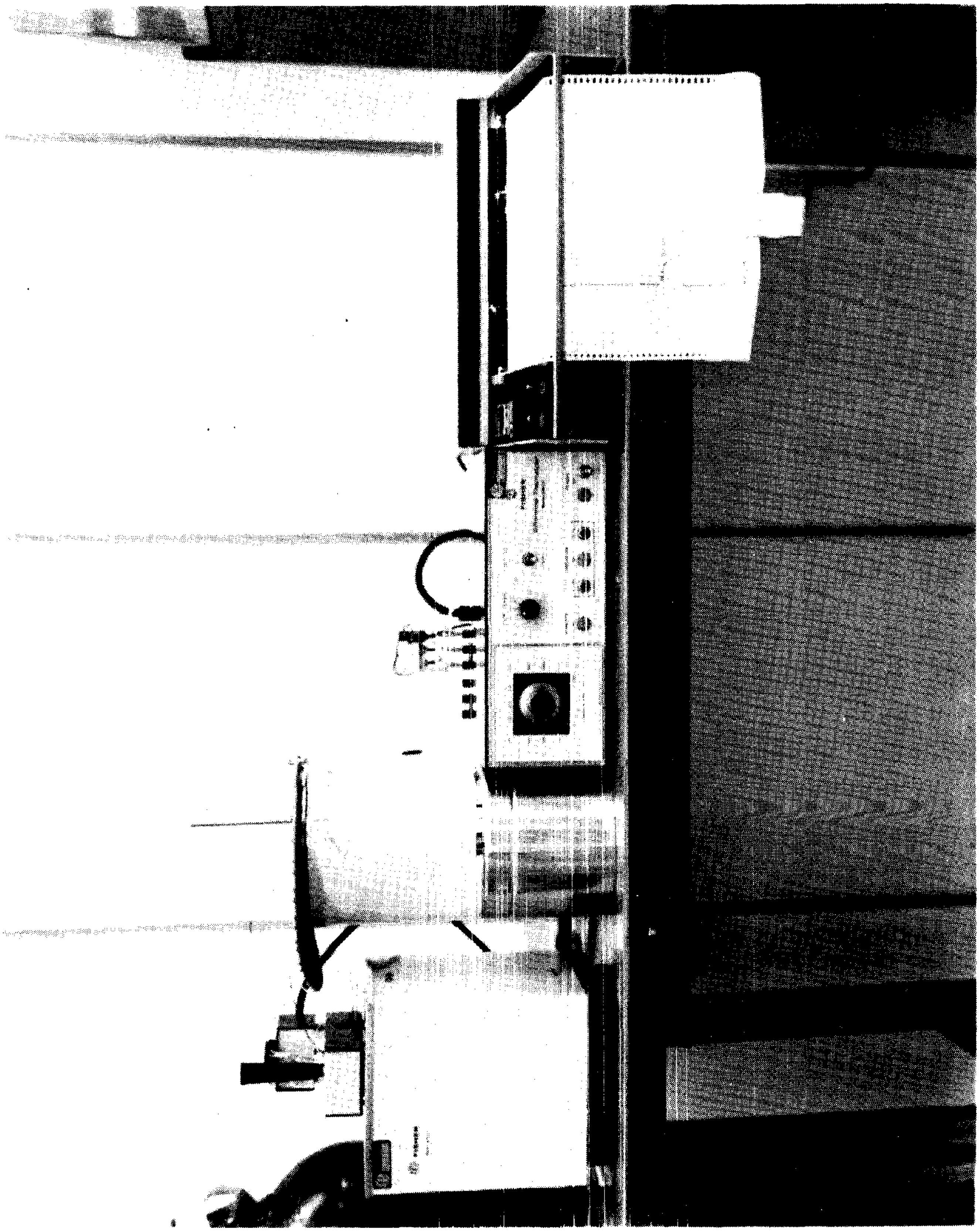

Figure 3-1. Differential Thermal Analysis Apparatus

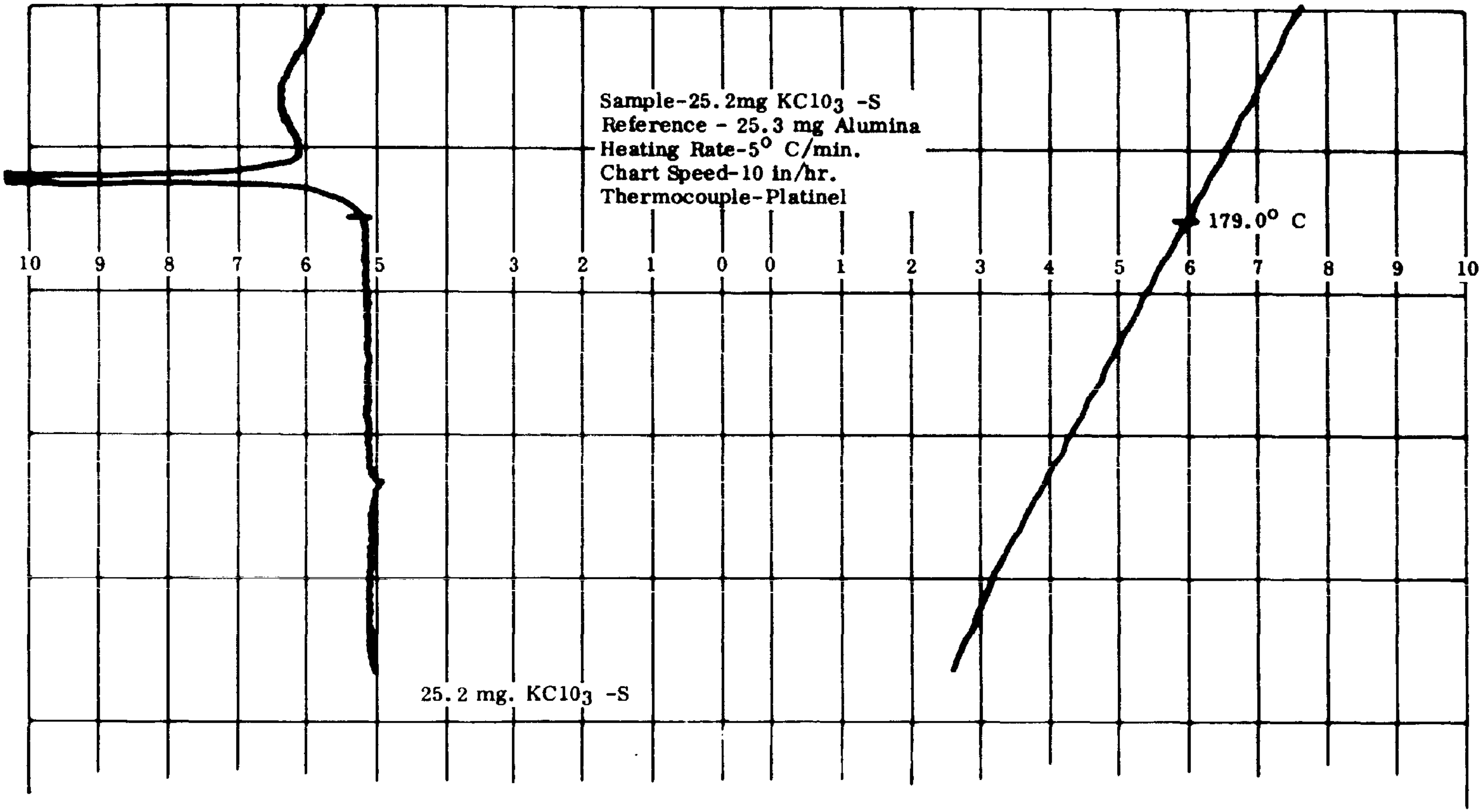

Figure 3-2. Differential Thermal Analysis Thermogram, Typical

3.2.2 CONCLUSIONS AND RECOMMENDATIONS

The DTA technique is recommended for the determination of ignition sensitivity for pyrotechnics. It would be difficult with the small amount of data available from these tests, to correlate a hazards classification with ignition temperature. There is enough of an indication that DTA sensitivity correlates with other sensitivities (see Table 5-1) to undertake additional research in this area.

3.3 INSTRUMENTED IMPACT TESTS

3.3.1 GENERAL

Based upon recommendations made in GE-MTSD-R-035, Pyrotechnic Hazards Classification and Evaluation Program, Phase I Final Report, May 1970, the Bureau of Explosives impact apparatus was instrumented in order to measure:

- Drop weight acceleration
- Input energy to the sample
- Sensitivity to initiation

Results of the measurements collected would enable calculations of:

- Dwell time (that time that the falling weight rested upon the sample)
- Terminal velocity (velocity at impact only)
- Time to reaction (velocity of the falling weight)

The tests as described below were conducted as part of the impact sensitivity test series for Picatinny Arsenal, Contract NAS8-25149 and reported in GE-MTSD Report Number R-056, dated March 26, 1971.

3.3.2 TEST SETUP

The instrumentation was comprised of a stationary set of electrical contacts attached to the support arm of the falling weight at one-inch intervals. A wiper was attached to the falling weight (see Figure 3-3). The output of the wiper was then fed to an oscillograph recorder (see the electrical schematic, Figure 3-4 and oscillograph, Figure 3-5). A strain gage was attached to the plunger to measure the force of the impact. This was fed to a bridge circuit/power supply and then to the oscillograph recorder (see Figure 3-6).

3.3.3 TEST RESULTS

Pyrotechnic mixtures and primary explosives were used to determine the validity of the instrumentation system. Specific tests were performed at different drop heights to determine dwell time, acceleration due to gravity for the falling weight and dwell time.

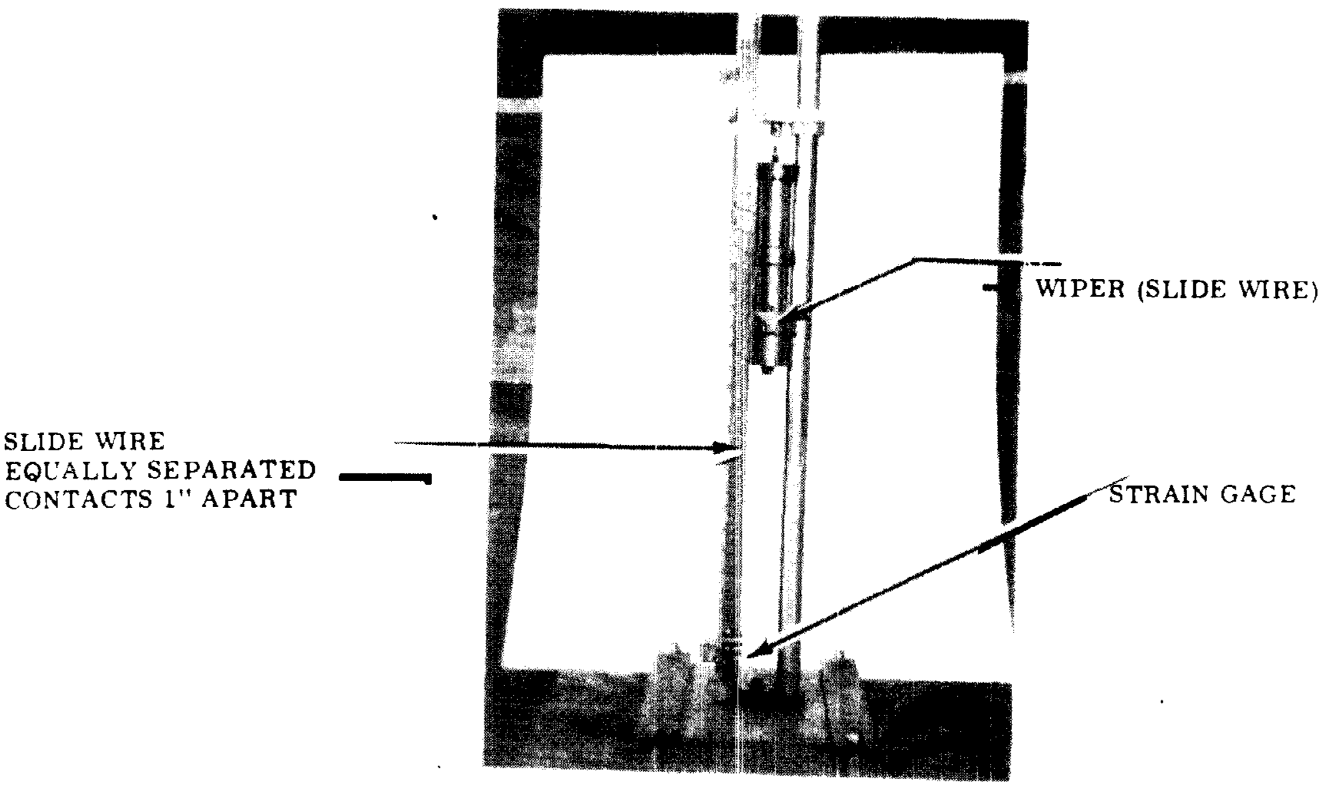

Figure 3-3. Test Configuration for Terminal Velocity and Dwell Time Measurements

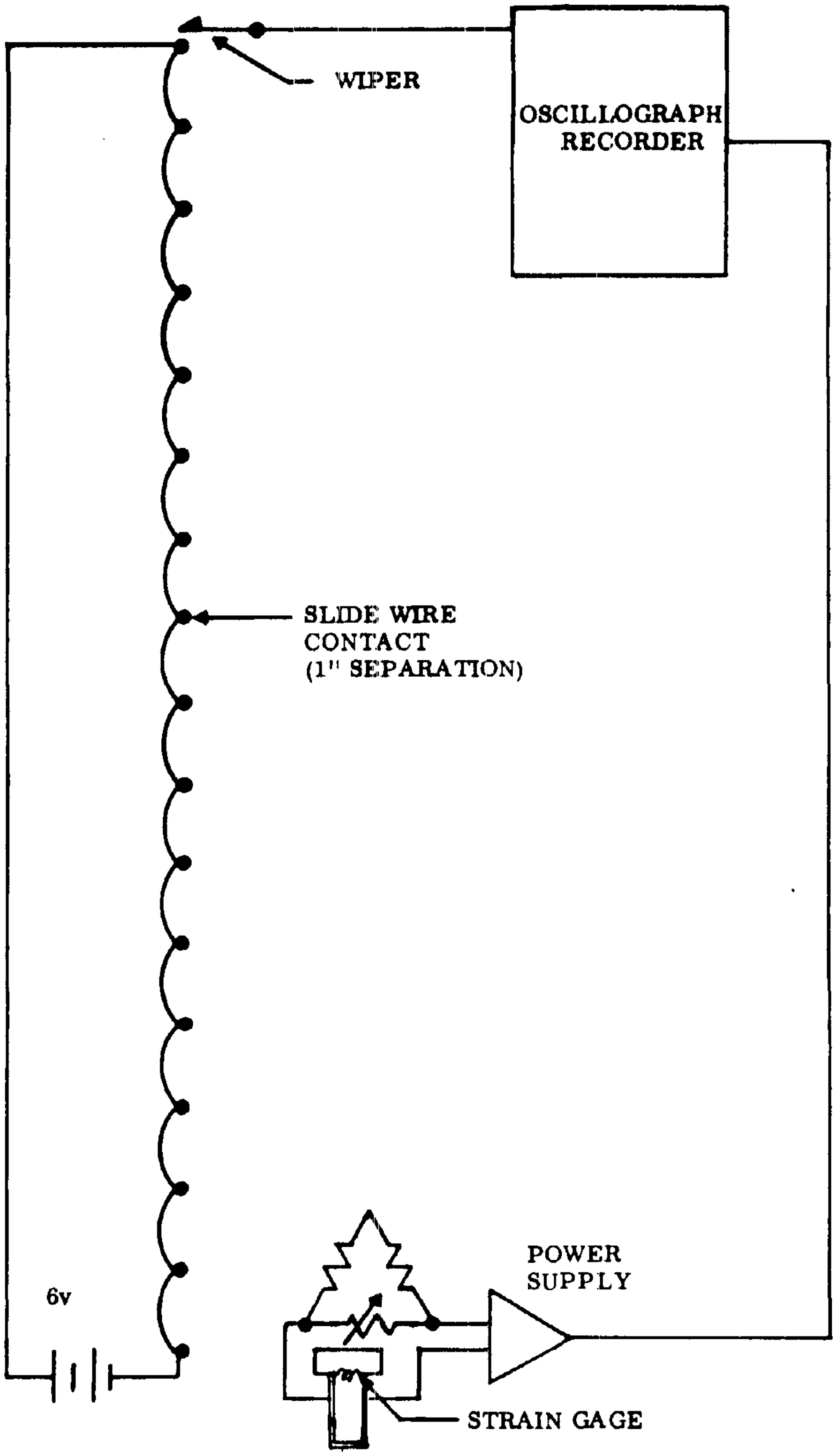

Figure 3-4. Electrical Schematic of Impact Instrumentation

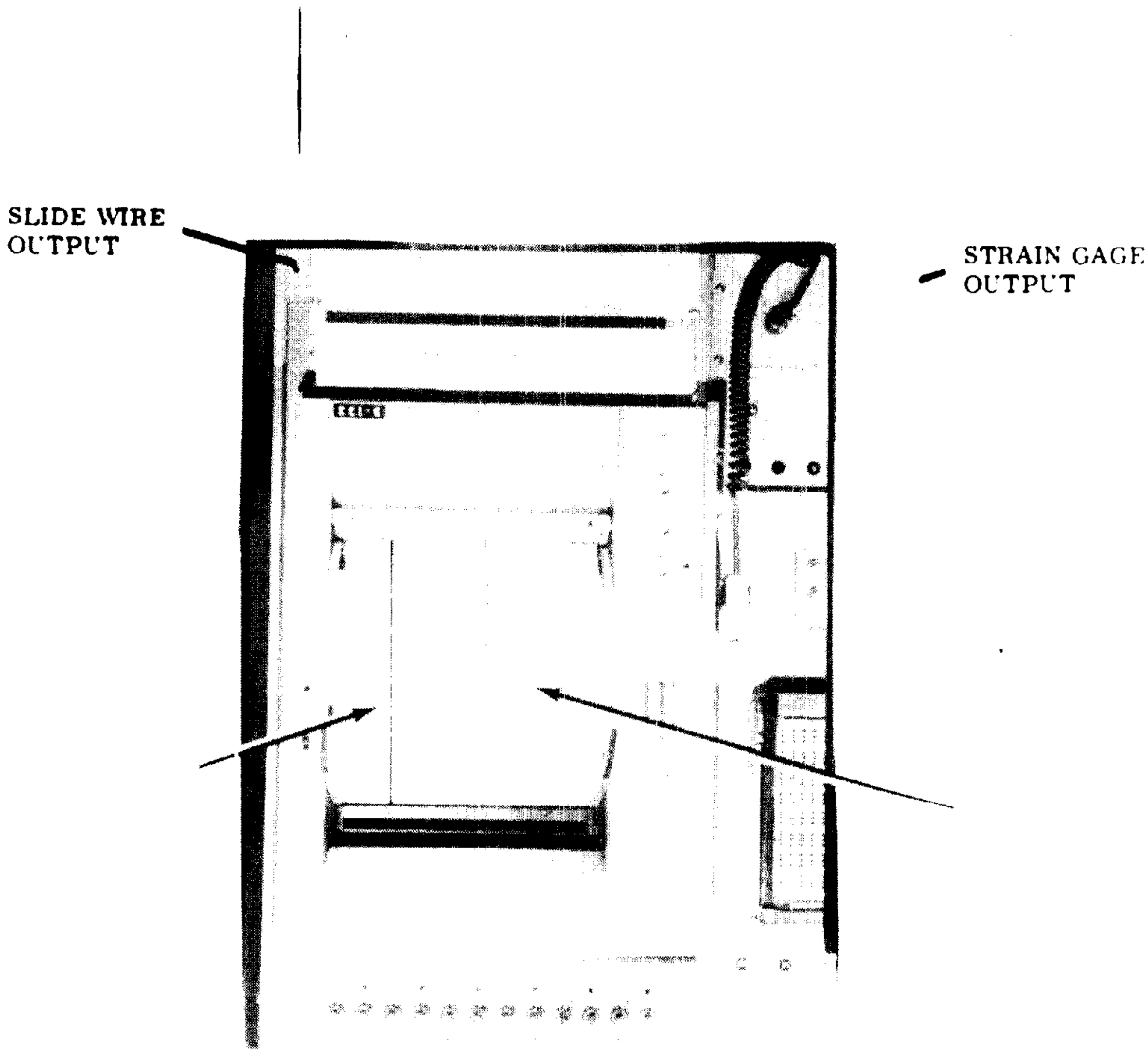

Figure 3-5. Oscillograph Used for Data Collection for Terminal Velocity and Dwell Time

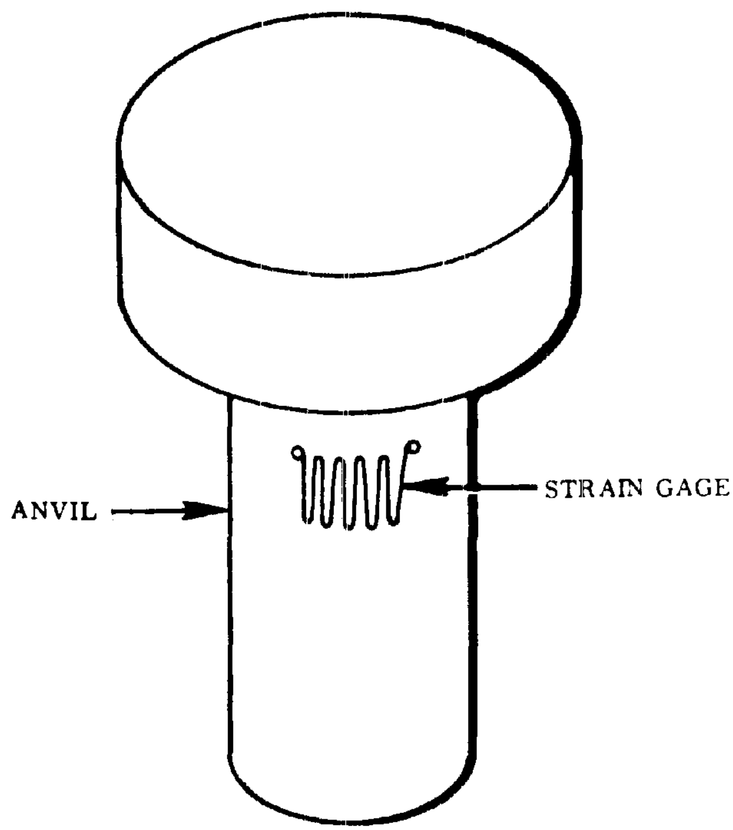

Figure 3-6. Typical Strain Gage Arrangement for Instrumented Impact Tests

3.3.4 TERMINAL VELOCITY

The terminal velocity was calculated for each drop height of (3-3/4, 4, 10, and 15 inches). This data is graphically displayed in Table 3-2 along with measured data from tests performed at these four heights. The inherent characteristics of the test apparatus may be depicted by the loss due to friction. Better correlation was shown at the 7- and 10-inch heights, but additional testing should be conducted to evaluate "operator" variances.

3.3.5 DWELL TIME

Dwell time was measured at each of the four drop heights in conjunction with velocity measurements and measures that time that the falling weights rest upon the plunger of the impact apparatus. The significance of the dwell time and the terminal velocity is indicative of the force applied to the sample material for any given height. This data is shown in Table 3-3.

3.3.6 ACCELERATION

Acceleration was measured at each drop height and is graphically shown in Figure 3-7. This curve shows the difference between the theoretical calculation and measured results. The results show that the least amount of deviation from the theoretical values was between 7 and 10 inches, whereas the greatest amount of deviation occurred at 3-3/4 and 15 inches, consistant with dwell time and terminal velocity data. These differences may also be attributed to the characteristics of the apparatus "operator" and the variances relatively small number of samples measured at each height. Efforts at elimination of operator effects, together with increased number of tests.

3.3.7 STRAIN GAGE

Strain gage was used to measure the force applied and also to see if it was possible to detect a difference between a non-explosive reaction and an explosive reaction. This is demonstrated in Figure 3-8 and 3-9. These oscillograph traces show that there is a distinct difference in an "explosive reaction" versus a "non-explosive" reaction. For each "explosive" reaction a multiple or jagged trace was observed, while for each "non-explosive" reaction, the trace was smooth (see Figures 3-8 and 3-9). Additionally, a decreased amplitude was recorded for explosive, as compared to non-explosive reactions, because of the phase relationship of the forces involved. This characteristic was also noted in observations of dwell time.

Another characteristic of the "explosive" trace was noted, namely that the strain gage output trace became negative after each reaction. Investigation showed that this result was a function of the strain gage mounting for this particular application, and does not necessarily constitute an identifying criteria.

In all cases, the reaction which occurred (explosive or non-explosive) was compared to the SSS (sight, smell, sound) method to confirm the validity of the strain gage system. For the tests conducted, the strain gage system was 100 percent reliable in predicting an "explosive" reaction. "Decomposition" reactions were not predictable and showed the same type of signature on the oscillograph trace as a "non-explosive" (or "no-reactive").

Table 3-2. Terminal Velocity of Impact Tester Falling Weight
(No Sample Used in Test)*

Height of Falling Weight	Calculated Velocity in feet/sec ($V_f^2 = V_o^2 + 2\ gh$)	Recorded Velocity in feet/sec ($V = V_o + gt$)	Apparent Loss Due to Friction feet/sec
15"	8.95	8.49	0.46
10"	7.40	7.10	.30
7"	6.10	5.82	.28
3-3/4"	4.48	3.68	0.80

*5 data points randomly selected for each height, from a total of 560 measurements

Table 3-3. Dwell Time of Falling Weight on Sample
(Random Samples and Reactions)*

Test Number	Drop Height 3-3/4" Dwell Time in msec	Drop Height 7" Dwell Time in msec	Drop Height 10" Dwell Time in msec	Drop Height 15" Dwell Time in msec
1	3.6	6.2	1.5	2.3
2	3.3	6.9	2.3	4.8
3	6.5	6.0	2.3	4.5
4	4.5	2.5	1.2	4.3
5	5.8	2.0	2.4	4.8
6	2.0	1.0	4.0	2.1
7	2.5	2.5	4.8	2.3
8	2.4	1.1	6.0	1.3
9	2.3	7.8	4.3	1.1
10	2.5	6.0	4.5	1.1
	$\bar{x} = 3.54$	$\bar{x} = 4.20$	$\bar{x} = 3.3$	$\bar{x} = 2.86$

*Includes both explosive and non-explosive reactions

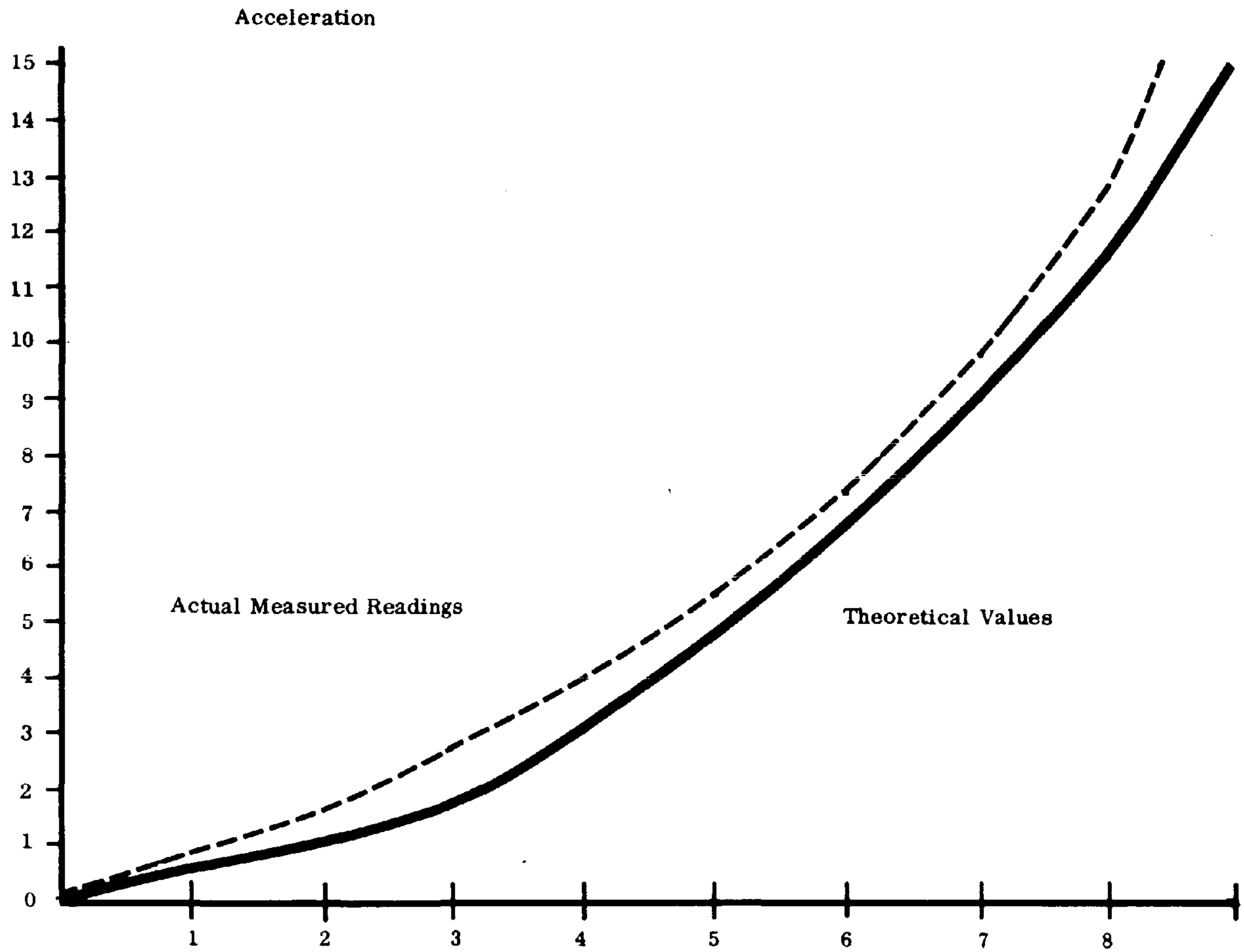

Figure 3-7. Acceleration of The Falling Weight

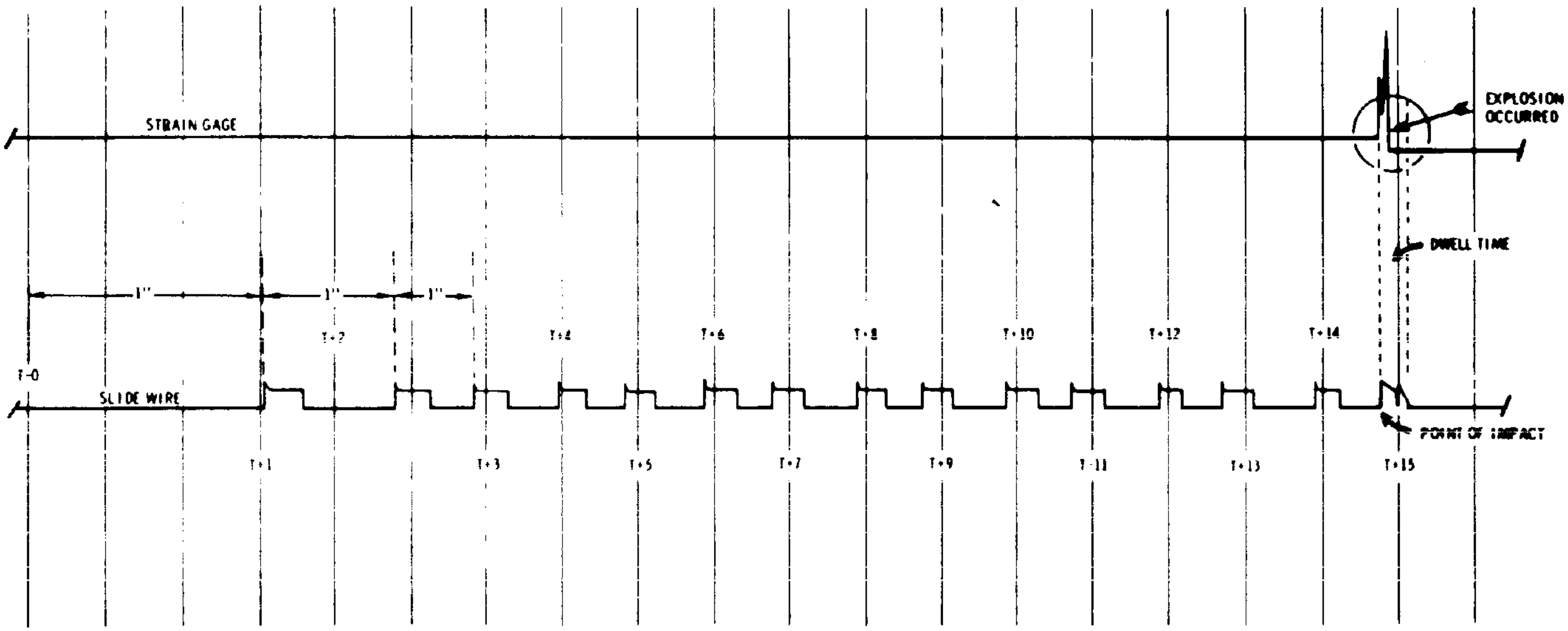

Figure 3-8. 15-Inch Impact Test Showing Strain Gage Trace for an Explosive Reaction

3.3.8 SUMMARY OF TEST RESULTS

The slidewire technique demonstrates that it is feasible to measure terminal velocity, acceleration, and dwell time of the Bureau of Explosives impact apparatus. These measurements determine the inherent characteristics of the impact apparatus and when coupled with the strain gage data (force applied and type of reaction) eliminates operator error in the interpretation of borderline reactions. The strain gage is able to detect a different reaction for a "non-explosive" event versus an "explosive" event. Further study and application of this technique are warranted in order that a statistical correlation with an acceptable degree of probability may be established. In other words, sufficient tests must be run with a variety of compositions to establish a characteristic signal or signature for each of the required parameters; i.e., "explosion, "decomposition," and "no reaction."

3.3.9 SAMPLE SIZE EVALUATION TESTS

Tests were also conducted under the Picatinny Arsenal impact sensitivity test program on varying sample sizes and at increasing drop heights. Sample sizes of 20, 30, 40, and 50 mg were tested at 3-3/4, 7, 10, and 15 inches. The significant finding from these tests was that for a given drop height, as the sample size increased the number of reactions decreased, presumably due to the cushioning effect of the sample material and the heat sink effect of the large mass. It was also found that the number of reactions for a given weight increased in direct proportion with increasing drop height. From this we may conclude that if larger sample sizes are used as recommended for a greater statistical validity, we must increase the drop heights proportonately.

3.4 ELECTROSTATICS TESTING

3.4.1 GENERAL

Electrostatics is the field of study that deals with phenomena due to attractions or repulsions of electric charges but not dependent upon their motion. The mechanisms involved in the process of developing the electric charges are still subject to further research even though many tests have been written on the science of electrostatics.

Electrostatics has to be considered as a potential hazard to pyrotechnic manufacturing, storage, and transportation, since the energy involved can cause ignition. Elimination of this energy is the problem to be solved when pyrotechnics are exposed to electrostatic charges. Because of the many variables and factors involved in electrostatics, each case where electrostatics may be a hazard to pyrotechnics is probably unique.

The primary point to consider is that of a spark occurring when an electrostatic charge is being created or neutralized. A spark produces heat, light, a small shock wave, and an electromagnetic field. It is the heat of the spark that is the most probable cause of ignition of pyrotechnics although the other forces can also cause ignition.

The electrostatic phenomena, theories, causes, prevention, characteristics, definitions and formula are detailed further in Appendix D.

3.4.2 TEST SETUP

In order to determine the minimum energy for spark ignition of a dust layer, a test setup such as that shown in Figure 3-10 was used. The electrical energy required for ignition of a layer of pyrotechnic powder is determined by discharging a condenser across a spark gap containing a layer of pyrotechnic material. The test setup consists of connecting the positive terminal of the condenser to a probe and the negative terminal to a sample cup.

3.4.3 TEST PROCEDURE

Preparations for testing was as follows:

a. Assemble the test equipment into the configuration shown in Figure 3-10.

b. Secure the specimen or components to be tested.

c. Ensure that all personnel within ten feet of the pyrotechnic test specimens are wearing safety glasses.

Actual testing proceeded as follows:

a. Verify that the high voltage power supply is off.

b. Place the test specimen in the test fixture (see Figure 3-10).

c. Ground the specimen as directed by the test conductor. Record the test configuration on the data sheet (Figure 3-11).

d. Turn on the high voltage power supply.

CAUTION

HIGH VOLTAGE. DURING THE REMAINING STEPS HIGH VOLTAGES WILL BE PRESENT. USE EXTREME CAUTION TO PREVENT ACCIDENTAL CONTACT WITH POINTS OF HIGH VOLTAGE.

e. With all output voltage switches to zero, turn the high voltage power switch on.

f. In the approximately five seconds between steps, advance the output voltage switches to the test voltage specified by the test conductor. Record the final voltage on the data sheet.

g. Using the control knob, lower the spark gap test aid probe to the sample until a spark occurs.

h. Return the spark gap test aid probe to its original position.

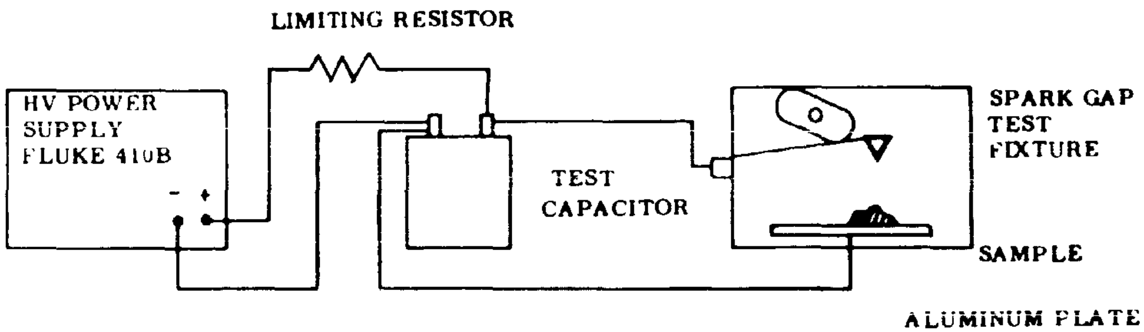

Figure 3-10. Electrostatic Ignition Susceptibility Test Setup

DATA SHEET

TEST NO.	TEST CONFIGURATION	VALUE OF CAPACITANCE (μf)	VOLTAGE LEVEL (VOLTS)	ENERGY $\frac{1}{2}CV^2$ (JOULES)	TEMPERATURE 67° / HUMIDITY 66% / DATE 10-8-70 — OBSERVATIONS AND COMMENTS
1	LY	.1	2000		Smoke
2	LY	.1	1900		Smoke
3	LY	.1	1800		Smoke
4	LY	.1	1700		Smoke
5	LY	.1	1600		Smoke
6	LY	.1	1500		Smoke
7	LY	.1	1500		Smoke
8	LY	.1	1400		No Smoke
9	LY	.1	1500		Smoke
10	LY	.1	1400		No Smoke
11	LY	.1	1500		Smoke
12	LY	.1	1400		Smoke
13	LY	.1	1300		Smoke

Figure 3-11. Electrostatic Ignition Susceptibility Test Data Sheet

i. Return the power supply high voltage output switches to zero.

j. Record observations and comments concerning the results of the test on the data sheet.

k. Upon completion of the test series, turn off the high voltage power supply.

l. Make the necessary calculations and complete the data sheet.

Electrostatic sensitivity tests were conducted as described above using four lactose base and four sulfur base colored smoke compositions in addition to the HC smoke composition. The resultant data transcribed from the data sheets is shown in Table 3-4. A standard statistical routine was used in treating the data. A typical mathematic solution is shown below for the Lactose Red pyrotechnic composition tested:

E_i (Ignition Energy) Joules	d_i Deviation	$(d_i)^2 \times 10^{-6}$
.242	-.006	36
.264	-.028	784
.288	-.052	2704
.269	-.028	784
.242	-.006	36
.210	.026	676
.210	.026	676
.210	.026	676
.242	-.006	36
.210	.026	676
.210	.026	676
2.592		7760

$$\text{Mean Energy Value} = E_m = \frac{1}{m} \cdot \sum_{i=1}^{n} \cdot E_i = .236 \text{ joules}$$

$$\text{Deviation} = d_i = E_i - E_m$$

$$\text{Standard Deviation} = \sigma = \left[\frac{1}{n-1} \cdot \sum_{i=1}^{n} (E_i - E_m)^2 \right]^{1/2}$$

$$= \left(\frac{7760}{10} \times 10^{-6}\right)^{1/2}$$

$$= .028 \text{ joules}$$

Table 3-4. Electrostatic Ignition Sensitivity Values for Selected Pyrotechnic Compositions

PYROTECHNIC COMPOSITION	IGNITION ENERGY (JOULES) E_m	STANDARD DEVIATION $\pm$ 1 (JOULES)
Lactose Yellow	.102	.005
Sulfur Yellow	.113	.018
Lactose Green	.121	.015
HC (White)	.122	.032
Sulfur Green	.131	.047
Sulfur Red	.154	.015
Sulfur Violet	.161	.019
Lactose Violet	.209	.062
Lactose Red	.236	.009
Mean E_m	.150	.046

At 1 σ, Mean E_m = .150 $\pm$ .046 = .104 joules to .196 joules

At 2 σ, Mean E_m = .150 $\pm$ .092 = .058 joules to .242 joules

At 3 σ, Mean E_m = .150 $\pm$ .138 = .012 joules to .288 joules

Assume all data points fall within Chauvent's criterion:

Thus for a probability of (1σ) 0.683; $E_m = .236 \pm .009$ joules

$$\sigma_m = \frac{\sigma}{\sqrt{n}} = \frac{.028}{3.66} \approx .009 \text{ joules}$$

For 2σ = .954; $E_m = .236 \pm .018$ joules

For 3σ = .997; $E_m = .236 \pm .027$ joules

Table 3-4 shows the resultant data for the nine samples tested. In all cases, 10 mg samples were tested. The energy value that produced a reaction, i.e., smoke or flame, was recorded for each test. Tests were run until 12 ignition energy values were obtained.

3.4.4 CONCLUSIONS AND RECOMMENDATIONS

As discussed previously in the section on DTA testing, the electrostatics data were inconclusive upon immediate examination. Some of the more generalized conclusions which may be drawn here are as follows:

- Considering the sample size and the test procedure, the ignition energy values are in a fairly tight range; i.e., .102 Joules for lactose yellow and .236 Joules for lactose red. Disregarding lactose red and lactose violet, the range narrows to .102 - .161 Joules.

  A man working in a normal working environment may develop a potential of 10,000 volts, and with an assumed capacitance of 200 picofarads, he has the capability of delivering .01 Joules of energy across a given air gap ($E = 1/2\ CV^2$). The value of .01 Joules is well below the range of values acquired for the electrostatic test series. However, transfer of body charge to an accumulating surface can generate charges which approach the threshold of ignition.

- A comparison of these data with other data will be made in succeeding sections in order to determine if a correlation is indicated; i.e., if the most electrostatic sensitive compound is also the most sensitive to DTA, impact, etc.

- Without additional data, refinement of technique and equipment, a recommendation of this test for hazards classification of pyrotechnics cannot be made. A standardized electrostatic sensitivity test for pyrotechnics should be developed.

3.5 HEAT OF COMBUSTION - BOMB CALORIMETER

3.5.1 GENERAL

Test samples of selected sample materials are burned in an oxygen filled metal "bomb" submerged in a measured quantity of water. By observing the rise in water temperature resulting from combustion of the sample, a calculation of the number of heat units (calories) liberated will be performed.

Standard test methods will be used with ASTM procedure D240-64, "Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter," as the prime reference.

The calculation of the gross heat of combustion (GHC) of the sample is based on the following equations:

$$\text{GHC (Btu. per lb)} = \frac{1.8\ (\text{two}-e_1-e_2-e_3)}{g} + 1.6\ (t_c - 25)$$

(Equation 2-1)

where: t = corrected temperature use, C^{o}, as calculated in Equation 2-2

t_c = maximum temperature, C^{o}, reached after firing corrected for thermometer errors

w = water equivalent of calorimeter

e_1 = corrections, calories, or HNO_3 formed (230 calories per gram)

e_2 = corrections, calories, of sulfur content (1300 calories per gram) for differences in heats of formation of sulfur dioxide and aqueous sulfuric acid. (This represents an additional correction as sulfuric acid has been calculated as nitric acid.)

e_3 = correction, calories, for iron firing wire (1600 calories per gram)

g = weight of sample in grams

The rise in temperature of the calorimeter water shall be corrected for loss and gain of heat as follows:

$$t = t_c - t_a - r_1\ (b-a) + r_2\ (c-b)$$

(Equation 2-2)

where: t = corrected temperature rise in C^{o}

a = time of firing

b = time when rise of temperature has reached six-tenths of total amount

c = time when temperature has reached a maximum after firing

t_a = temperature at time a, corrected for thermometer errors

t_c = temperature at time c, corrected for thermometer errors

r_1 = rate of temperature rise in C^{o} per minute for 5 minutes before firing

r_2 = rate of temperature drop in C^o per minute for 5 minutes after reaching the maximum temperature

b-a and c-b = time intervals expressed in minutes

3.5.2 HEAT OF COMBUSTION DATA

Results from the Parr Bomb tests for the pyrotechnic samples are tabulated as follows:

Sulfur Red	2282 calories/grams
Sulfur Violet	2294 calories/grams
Sulfur Yellow	2275 calories/grams
Sulfur Green	2487 calories/grams
Lactose Red	2988 calories/grams
Lactose Violet	2345 calories/grams
Lactose Yellow	2763 calories/grams
Lactose Green	2960 calories/grams
HC White Smoke	939 calories/grams
Fuel Mix	1000 calories/grams

The values expressed are based on the addition of oxygen at 5 atmospheres pressure which was necessary to assure total combustion of the sample.

Computer processing was used to calculate the heat of combustion values. The use of the heat of combustion technique to explore the pyrotechnic material reaction characteristics should be researched further with potential applications wherein the actual pressure rise and burn rate of the material are determined. Initial testing of the pyrotechnic materials using a modified "Parr Bomb" to measure the internal pressure rise showed that there are both discrete pressure and burn rate values for the various pyrotechnic compositions.

This is still another area for further research in order to determine the applicability of this test to hazards classification and evaluation.

3.6 PHASE I TEST PROGRAM

3.6.1 INTRODUCTION

In the Phase I Final Report (GE-MTSD Report Number R-035, dated May 1970), certain conclusions were drawn and recommendations were made as a result of TB 700-2 testing and TNT equivalency testing of a number of pyrotechnic granular bulk compositions and end item munitions. Table 3-5 identifies, by U.S. Army drawing number, the materials tested under Phase I. Following Table 3-5 are Tables 3-6 through 3-14 which summarize the TB 700-2 test results as well as the TNT equivalency test results. In succeeding paragraphs are the conclusions and recommendations.

3.6.2 PHASE I CONCLUSION

3.6.2.1 General

When Phase I testing was completed and the test results (shown above) analyzed, certain specific and generalized conclusions were drawn. Based on these conclusions, appropriate recommendations were made. The conclusions are presented in this section, followed by the recommendations under paragraph 3.6.3. It must be kept in mind that these conclusions and recommendations were made on the basis that TB 700-2 did not provide relative sensitivity data, only "go - no-go"/yes - no type answers. As discussed earlier in Sections 1 and 2, TB 700-2 probably provides the one basic answer that it was intended to provide; i.e., the material (pyrotechnics) are Class 2 (fire hazards) or Class 7 (mass detonating).

In addition, these conclusions and recommendations are directed to suggestions for improving the TB 700-2 techniques, procedures, hardware, and instrumentation. Phase III is directed to that other facet of a TB 700-2 type specification which provides data upon which hazards can be evaluated in terms of initiation sensitivity, ease of communication, and ability of transition from a low order reaction to a detonation. These criteria permit cost effective design criteria to be developed for manufacturing, handling, storage and transportation equipment and/or facilities.

3.6.2.2 Detonation Test

All testing Phase I to date confirms the desirability of appropriate revisions of TB 700-2 for application to pyrotechnic compounds. For example, the Standard Detonation Test does not lend itself to meaningful testing and evaluation of granular materials. Additionally, the testing procedure does not provide for containment of the granular sample nor for standard compression, tamping, or confinement of the material. During the test program, laboratory filter paper was used to construct a cube shaped box to hold the required 2-inch cube sample.

It was found that in the case of pyrotechnic materials, mushrooming of the lead cylinder did not occur. If it had occurred, there was no provision in TB 700-2 to describe whether the "mushrooming" was 1/16 inches or 2 inches, etc. In an effort to detect any minute distortions in the lead cylinders, a "go-no-go" gage with 1/16 inch clearance was constructed to check for "mushrooming."

Table 3-5. Ignition and Unconfined Burning Test Results

Sample Material	Single Cube Test			Single Cube Test			Multiple Cube Test		
	Exploded		Burning Time Seconds	Exploded		Burning Time Seconds	Exploded		Burning Time Seconds
	Yes	No		Yes	No		Yes	No	
SG 3-69-1		X	29		X	31		X	55
SR 3-69-1		X	40		x	39.5		X	65
SY 3-69-1		X	35		X	36		X	56
SV 3-69-1		X	22		X	25		X	30
LG 3-69-1		X	35		X	33		X	36
LR 3-69-1		X	16		X	21.5		X	28.2
LY 3-69-1		X	26		X	25		X	36
LV 3-69-1		X	11		X	10		X	12.2
FM 3-69-1		X	4		X	6		X	6.4
HC 3-69-1		X	7 Min. * 18 Sec.		X	4 Min. * 8 Sec.		X	4 Min. * 8 Sec.
CS 7-752		X	18		X	13		X	17
SM II		X	10		X	10		X	12.6
SM III		X	22.8		X	22.4		X	38.6
SM V		X **	3.1		X**	3.8		X	4.8
SM VI		X	12.4		X	15.6		X	20.1
SM XII	X***		0.8	X ****		0.8	X *****		1.6
SM XXV		X	5.0		X	5.0		X	5.8
FF VII		X	6.5		X	5.0		X	7.0

*Burn time of kerosene - sawdust mixture - HC smoke mixture did not burn except for small percentage of outer crust

**Deflagration occurred, particle found 10 feet from point of ignition

***Deflagration (per TB 700-2) - some particles left pan and were scattered unburned

****Deflagration (per TB 700-2) caused sawdust to leave the pan and extinguish the fire

*****Deflagration (per TB 700-2) - some unburned particles found 25 feet from pan

Table 3-6. Thermal Stability Test Material and Results

SAMPLE MATERIAL	EXPLOSION		IGNITION		CHANGE IN CONFIGURATION	
	YES	NO	YES	NO	YES	NO
SG 3-69-1		X		X		X
SR 3-69-1		X		X		X
SY 3-69-1		X		X		X
SV 3-69-1		X		X		X
LG 3-69-1		X		X		X
LR 3-69-1		X		X		X
LY 3-69-1		X		X		X
LV 3-69-1		X		X		X
FM 3-69-1		X		X		X
HC 3-69-1*		X		X	X	
CS T-752		X		X		X
SM II		X		X		X
SM III		X		X		X
SM V**		X		X	X	
SM VI		X		X		X
SM XII		X		X		X
SM XXV		X		X		X
FF VII		X		X		X

* Sample HC 3-69-1 Lost 100 Grams Total Weight - retested with same result

** Sample SM V Lost 19.5 Grams Total Weight - retested with same result

Table 3-7. Card Gap Test Results

SAMPLE MATERIAL	DETONATION		50% VALUE	NUMBER OF CARDS
	YES	NO		
SG 3-69-1		X	N/A	NONE
SR 3-69-1		X	N/A	NONE
SY 3-69-1		X	N/A	NONE
SV 3-69-1		X	N/A	NONE
LG 3-69-1		X	N/A	NONE
LR 3-69-1		X	N/A	NONE
LY 3-69-1		X	N/A	NONE
LV 3-69-1		X	N/A	NONE
FM 3-69-1		X	N/A	NONE
HC 3-69-1.		X	N/A	NONE
CS T-752		X	N/A	NONE
SM II		X	N/A	NONE
SM III		X	N/A	NONE
SM V		X	N/A	NONE
SM VI		X	N/A	NONE
SM XII		X	N/A	NONE
SM XXV		X	N/A	NONE
FF VII		X	N/A	NONE

Table 3-8. Detonation Test Results

Sample Material	EXPLODED		BURNED		FRAGMENTED *	
	Yes	No	Yes	No	Yes	No
SG 3-69-1		X		X		X
SR 3-69-1		X		X		X
SY 3-69-1		X		X		X
SV 3-69-1		X		X		X
LG 3-69-1		X		X		X
LR 3-69-1		X		X		X
LY 3-69-1		X		X		X
LV 3-69-1		X		X		X
FM 3-69-1		X		X		X
HC 3-69-1		X		X		X
CS 7-752		X	X			X
SM II		X	X			X
SM III		X	X			X
SM V		X	X			X
SM VI		X	X			X
SM XII		X	X			X
SM XXV		X	X			X
FF VII		X	X			X

* Fragmentation of the granular bulk materials tested is defined as the dispersion of the materials resulting from the explosive action of a No. 8 blasting cap.

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Table 3-9. Impact Sensitivity Rest Results

SAMPLE MATERIAL	3 3/4" DROP TEST			10 " DROP TEST		
	Explosion	Decomposition	No Reaction	Explosion	Decomposition	No Reaction
SG 3-69-1	- 0 -	- 0 -	- 10 -	- 0 -	- 5 -	- 5 -
SR 3-69-1	- 0 -	- 0 -	- 10 -	- 0 -	- 6 -	- 4 -
SY 3-69-1	- 0 -	- 0 -	- 10 -	- 0 -	- 5 -	- 5 -
SV 3-69-1	- 0 -	- 2 -	- 8 -	- 2 -	- 5 -	- 3 -
SV 3-69-1	- 0 -	- 0 -	- 0 -	- 0 -	- 8 -	- 2 -
Medium	- 0 -	- 0 -	- 0 -	- 2 -	- 7 -	- 1 -
Cold	- 0 -	- 0 -	- 0 -	- 1 -	- 6 -	- 3 -
LG 3-69-1	- 0 -	- 0 -	- 10 -	- 0 -	- 1 -	- 9 -
LR 3-69-1	- 0 -	- 0 -	- 10 -	- 0 -	- 1 -	- 9 -
LY 3-69-1	- 0 -	- 0 -	- 10 -	- 1 -	- 3 -	- 6 -
LY 3-69-1	- 0 -	- 0 -	- 0 -	- 0 -	- 4 -	- 6 -
Medium	- 0 -	- 0 -	- 0 -	- 0 -	- 4 -	- 6 -
Cold	- 0 -	- 0 -	- 0 -	- 0 -	- 2 -	- 3 -
LV 3-69-1	- 0 -	- 0 -	- 10 -	- 1 -	- 2 -	- 7 -
LV 3-69-1	- 0 -	- 0 -	- 0 -	- 0 -	- 2 -	- 3 -
Cold	- 0 -	- 0 -	- 0 -	- 0 -	- 2 -	- 8 -
FM 3-69-1	- 0 -	- 0 -	- 10 -	- 1 -	- 7 -	- 2 -
HC 3-69-1	- 0 -	- 0 -	- 10 -	- 0 -	- 0 -	- 10 -
CS T-752	- 0 -	- 0 -	- 10 -	- 0 -	- 0 -	- 10 -
SM II	- 0 -	- 0 -	- 10 -	- 0 -	- 0 -	- 10 -
SM III	- 0 -	- 0 -	- 10 -	- 0 -	- 0 -	- 10 -
SM V	- 0 -	- 0 -	- 10 -	- 0 -	- 0 -	- 10 -
SM VI	- 0 -	- 0 -	- 10 -	- 9 -	- 0 -	- 1 -
SM XII	- 0 -	- 0 -	- 10 -	- 2 -	- 5 -	- 3 -
SM XXV	- 0 -	- 0 -	- 10 -	- 0 -	- 0 -	- 10 -
FF VII	- 0 -	- 0 -	- 10 -	- 0 -	- 0 -	- 10 -

Table 3-10. End Item Tests

SAMPLE MATERIAL	TYPE OF TEST					
	DETONATION TEST A		DETONATION TEST B		EXTERNAL HEAT TEST C	
	PROPAGATION		PROPAGATION		EXPLOSION	
	Yes	No	Yes	No	Yes	No
M-18 Red		X	N/A	N/A		X
M-18 Green		X	N/A	N/A		X
M-18 Yellow		X	N/A	N/A		X
M-18 Violet		X	N/A	N/A		X
M-18 HC (White)		X	N/A	N/A		X
105 MM Canister HC (White)	X		X			X

Table 3-11. TB 700-2 End Item Tests

TEST RUNS	MATERIAL	AVERAGE TIME TO REACTION	AVERAGE REACTION TIME	REMARKS
		TEST A		
5	HC Smoke Grenade AN-M8 FSN 1330-219-8511	3 Seconds	4.5 Minutes	No Propagation
5	Violet Smoke M-18 Grenade FSN 1330-289-6852	5 Seconds	1.4 Minutes	No Propagation
5	Red Smoke M-18 Grenade FSN 1330-289-6852-16950	6 Seconds	2.5 Minutes	No Propagation
5	Yellow Smoke M-18 Grenade FSN 1330-289-6854-6945	Smoke at Ignition	1.1 Minutes	No Propagation
5	Green Smoke M-18 Grenade FSN 1330-289-6851-6940	6 Seconds	1.1 Minutes	No Propagation
1	HC Smoke (105M/M canister) FSN 1315-383-3889 (C396)	less than 2 seconds	150 Seconds	Limited propagation (to one other item)
1	HC Smoke (105M/M canister) FSN 1315-383-3889 (C396)	less than 2 seconds	150 Seconds	Profuse burning and jetting through 12 minutes secondary reaction after 25 minutes resulting in complete destruction of all canisters by 35 minutes no explosive dispersal of canisters
		TEST B		
1	HC Smoke (105M/M canister) FSN 1315-383-3889 (C396)	30 Seconds	14 Minutes	Total destruction - both containers no explosive dispersal
		TEST C		
1	HC Smoke (105M/M canister) FSN 1315-383-3889 (C396)	6 Minutes, 25 Seconds	13 Minutes 35 Seconds	Total destruction minor explosive dispersal (to 8' radius)
1	HC Smoke Grenade AN-M8 FSN 1330-219-857 Lot No. 2014-85-1077	12 Minutes	47 Minutes	Total destruction, scattering of grenades and fragments
1	Violet Smoke M-18 Grenade FSN 1330-289-6852 Lot No. 2044-75-1013	24 Minutes	31 Minutes	Total destruction, no scattering or fragments
1	Red Smoke M-18 Grenade FSN 1330-289-6852-6950 Lot No. PBA-40-33	13 Minutes	35 Minutes	Total destruction, no scattering or fragments
1	Yellow Smoke M-18 Grenade FSN 1330-289-6854-6945 Lot No. PBA-20-81	28 Minutes	58 Minutes	Total destruction, no scattering or fragments
1	Green Smoke M-18 Grenade FSN 1330-289-6851-6980 Lot No. DWG. 1-124	13 Minutes	47 Minutes	Total destruction, no scattering or fragments

Table 3-12. Summary of Probable Classifications

	DETONATION	IGNITION & UNCONFINED BURN	THERMAL STABILITY	IMPACT SENSITIVITY	CARD GAP	TB 700-2 CLASSIFICATION
LV 3-69-1	Class 2	Class 2	Class 2	Class 7	Class 2	Class 7
LY 3-69-1	Class 2	Class 2	Class 2	Class 7	Class 2	Class 7
LR 3-69-1	Class 2	Class 2	Class 2	Class 2	Class 2	Class 2
LG 3-69-1	Class 2	Class 2	Class 2	Class 2	Class 2	Class 2
SV 3-69-1	Class 2	Class 2	Class 2	Class 7	Class 2	Class 7
SY 3-69-1	Class 2	Class 2	Class 2	Class 2	Class 2	Class 2
SR 3-69-1	Class 2	Class 2	Class 2	Class 2	Class 2	Class 2
SG 3-69-1	Class 2	Class 2	Class 2	Class 2	Class 2	Class 2
HC 3-69-1	Class 2	Class 2	Class 2	Class 2	Class 2	Class 2
CS T-752	Class 2	Class 2	Class 2	Class 2	Class 2	Class 2
FM 3-69-1	Class 2	Class 2	Class 2	Class 7	Class 2	Class 7
SM II	Class 2	Class 2	Class 2	Class 2	Class 2	Class 2
SM III	Class 2	Class 2	Class 2	Class 2	Class 2	Class 2
SM V	Class 2	Class 2	Class 2	Class 2	Class 2	Class 2
SM VI	Class 2	Class 2	Class 2	Class 7	Class 2	Class 7
SM XII	Class 2	Class 2	Class 2	Class 7	Class 2	Class 7
SM XXV	Class 2	Class 2	Class 2	Class 2	Class 2	Class 2
FF VII	Class 2	Class 2	Class 2	Class 2	Class 2	Class 2

Table 3-13. Summary of Pyrotechnic Test Material TNT Equivalencies

Shown are the Mean Values and the Mean of the Means.

Sample Material	Number of Tests	% TNT Based on Mean Peak Pressure PSIG	Standard Deviation (n-1)	% TNT Based on Mean Impulse (PSI - Millisec.)	Standard Deviation (n-1)
SG 3-69-1	1	3.21	± 2.03	0.33	± 0.23
	2	3.67	± 1.21	0.43	± 0.27
	3	3.67	± 1.39	0.95	± 0.52
	Mean	4.30	± 1.51	0.57	± 0.33
SR 3-69-1	1	6.88	± 5.79	1.64	± 1.89
	2	2.64	± 0.90	0.27	± 0.22
	3	2.85	± 1.05	0.27	± 0.12
	4	4.44	± 1.74	0.69	± 0.33
	5	9.93	± 0.58	2.88	± 0.91
	Mean	5.35	± 3.07	1.11	± 1.04
SY 3-69-1	1	4.31	± 0.47	0.94	± 0.27
	2	4.44	± 0.72	1.55	± 0.65
	3	2.40	± 0.78	0.28	± 0.20
	Mean	3.72	± 1.14	0.92	± 0.63
SV 3-69-1	1	6.15	± 2.59	0.64	± 0.34
	2	7.43	± 1.35	1.87	± 0.58
	3	6.02	± 0.69	1.27	± 0.27
	Mean	6.53	± 0.78	1.26	± 0.62
LG 3-69-1	1	6.16	± 1.02	1.28	± 0.46
	2	5.70	± 1.09	0.67	± 0.12
	3	7.08	± 1.62	1.02	± 0.37
	Mean	6.31	± 0.71	0.99	± 0.31
LR 3-69-1	1	6.56	2.24	0.99	0.18
	2	5.80	0.25	0.54	0.10
	3	3.83	1.23	0.34	0.16
	Mean	5.40	1.41	0.62	0.33
LY 3-69-1	1	9.71	2.98	2.62	1.28
	2	8.26	1.11	1.75	0.39
	3	4.81	0.75	0.57	0.19
	4	5.67	3.07	0.73	0.55
	Mean	7.11	2.27	1.42	0.96
LV 3-69-1	1	5.70	1.09	0.91	0.33
	2	4.82	2.50	0.81	0.71
	3	3.65	1.84	0.32	0.20
	Mean	4.72	1.03	0.68	0.31
FM 3-69-1	1	10.42	1.49	2.90	0.83
	2	8.99	0.88	3.03	0.73
	3	10.83	2.05	3.00	1.05
	4	13.27	1.19	4.35	0.73
	Mean	10.88	1.78	3.32	0.69
CS T-752	1	10.16	3.79	2.92	1.31
	2	10.62	3.06	2.33	0.54
	3	10.62	1.78	2.96	0.69
	4	10.02	1.27	3.56	0.64
	Mean	10.38	0.31	2.94	0.50

Table 3-14. Test Materials

BULK COMPOUNDS		
Sulfur Green	Lot # 3-69-1	Drawing # B143-2-1
Sulfur Red	Lot # 3-69-1	Drawing # B143-3-1
Sulfur Yellow	Lot # 3-69-1	Drawing # B143-4-1
Sulfur Violet	Lot # 3-69-1	Drawing # B143-5-1
Lactose Green	Lot # 3-69-1	Drawing # B143-2-6
Lactose Red	Lot # 3-69-1	Drawing # B143-3-7
Lactose Yellow	Lot # 3-69-1	Drawing # B143-4-7
Lactose Violet	Lot # 3-69-1	Drawing # B143-5-2
Fuel Mix	Lot # 3-69-1	Drawing # B143-10-1
HC Smoke Mix	Lot # 3-69-1	Drawing # B143-1-1
Pure CS Mix	T-752	
STARTER MIXES		
Starter Mix XII (Wet Base)		Drawing # B143-7-1
Starter Mix VI (Wet Base)		Drawing # B143-7-3
Starter Mix XXV (Wet Base)		Drawing # B143-7-4
Starter Mix II (Dry)		Drawing # B143-7-5
Starter Mix III (Dry)		Drawing # B143-7-6
Starter Mix V (Wet Base)		Drawing # B143-7-9
END ITEMS		
AN-M8 Grenade, HC Smoke		FSN 1330-219-8511
M-18 Grenade, Violet Smoke		FSN 1330-289-6852
M-18 Grenade, Red Smoke		FSN 1330-289-6852-16950
M-18 Grenade, Yellow Smoke		FSN 1330-289-6854-6945
M-18 Grenade, Green Smoke		FSN 1330-289-6851-6940
105 mm HC Smoke Canisters		FSN 1315-383-3889 (396)
	ADDITIONAL SAMPLES	
BULK COMPOUNDS		
Lactose Yellow	Lot # 2-69-2	
Lactose Yellow	Lot # 3-69-3	
Sulfur Violet	Lot # 3-69-4	
Mix # 13		
STARTER MIXES		
First Fire VII		Drawing # C143-8-2

To answer the question as to whether the sample "fragmented," it was found necessary to supply a footnote to Form AG0793/A to explain that the action of the blasting cap "scattered" rather than fragmented the sample material.

3.6.2.3 Ignition and Unconfined Burning Test

The observed effects of minimal scattering and complete burning of the sample material indicates only that the pyrotechnic material performs the function it is generally intended to perform, i.e., burn at a designed rate. Any other use of the test is inconclusive since TB 700-2 does not contain criteria or requirements for the burning rate; therefore, there is no apparent relationship between burning rate and classification.

Again the problem exists in the preparation of a typical granular sample for testing using the 2-inch cube criteria. The specification should provide for granular bulk samples as well as consolidated samples. It is apparent that the specification is written for a typical high explosive or propellant which is generally a solid material that can be cut or machined into the required 2-inch cube.

3.6.2.4 Thermal Stability Test

It is difficult to ascertain from the small number of pyrotechnic materials that were subjected to the thermal stability test whether or not the test provides conclusive data with respect to these materials. The only positive results obtained from the 11 smoke sample compounds and seven starter mixes was a "change in configuration" in the HC smoke mix and Starter Mix V caused by a loss in volatile chemicals. The change was actually a change in weight and a slight reduction in the size of the sample.

Although the sample cube was provided with a thermocouple, no unusual temperature deviations were observed on the strip chart recorder data sheets. Dual thermocouple should be imperative for any type of material where an exothermic or endothermic reaction might be expected to occur.

3.6.2.5 Impact Sensitivity Test

The conclusions derived during this test program relative to the impact sensitivity test were made with respect to the factors of blending, screening, and mixing of the sample as a primary consideration. The size of sample and the capability to duplicate the identical mixture of a particular sample during the test sequence is unpredictable and warrants further examination. It is safe to assume that the probability of drawing a sample representative of the total mix or lot (bulk) each time a 10 milligram sample is taken is infinitesimally low. Increasing the size of the sample tested may increase the validity of the results.

Statistically the results taken from a 20 test drop sampling are inconclusive. The population (quantity) of tests should be increased to permit better statistical correlation. It would also be advisable to examine this test in terms of degree of sensitivity by performing the test drop at an increasing height until detonation is exhibited or a maximum limit is reached. Computation could then be oriented to a degree of sensitivity.

3.6.2.6 Card Gap Test

3.6.2.6.1 General

The card gap test, by observation of test results performed on pyrotechnics, is another in the series of "go-no-go" tests characteristic of the TB 700-2 specification. The violent reaction of the two pentolite pellets, as demonstrated by the fragmentation of the card gap tube and the hole punched in the witness plate (when fired independent of any sample material), makes measurement of any reaction less than a detonation by the donor sample difficult. The fact that the witness plate is only deformed in the pyrotechnic tests tends to confirm the relative stability of the pyrotechnic and would indicate an attenuation of the pentolite reaction. The difficulty in relating deformation of the witness plate to other factors, such as TNT equivalency, is further proof of the relative stability of the samples. The slight variance in the recorded overpressure and impulse data from the instrumented card gap tests when compared to the open air bursts of pentolite indicates that there is little additive reaction from the sample to the pentolite.

The "go-no-go" characteristics of the card gap test warrants further examination with respect to its use as a means of determining degree of sensitivity. When testing high explosives, the introduction of cellulose acetate cards between the sample and the pentolite does offer a sensitivity range computation capability. Without detonation, as occurs with the types of pyrotechnics tested in this program, the sensitivity measurement is not possible.

3.6.2.6.2 Witness Plate Material

After performing the special tests with the different witness plate materials, it must be concluded that the specification requirements with regard to the steel plate must be more explicitly defined. If, in fact a witness plate can shatter and void a test, a witness plate could also fail to produce valid "go-no-go" results due to variations in the properties of the steel within the specification.

3.6.2.6.3 Witness Plate Volumetric and Deformation Measurement

Based on the relatively limited potential energy range of materials tested, the work performed in linear and volumetric measurement of card gap witness plat deformation was rather inconclusive. An effort to correlate deformation data with TNT equivalency with little or no conclusions obtained was made. Until more exact measurement techniques are employed, such as burning rate probes and pressure transducers inside the pipe, the slight variations in energy release in the card gap configuration will be difficult to determine.

3.6.2.6.4 Orientation

Card gap tests were fired in a 90° and 180° orientation from that specified by TB 700-2 to determine primarily the effects on the blast pressure data. It was determined that the overpressure distortions caused by the previously discussed asymmetric rupturing of the sample pipe were only exaggerated by reorientation. It was also found that the inverted or the horizontal card gap test setup only resulted in difficult recovery of the witness plate. An additional hazard is also introduced into the test program caused by a large size fragment in the form of the witness plate.

3.6.2.6.5 Inert Sample Tests

Card gap tests run with an empty sample tube and the normal configuration showed greater plate distortion than any of the pyrotechnic samples tested. Conversely, ordinary sand tested in the card gap configuration exhibited little or no distortion of the plate. It can be concluded from these results that the pyrotechnic material only serves to attenuate the blast pressure wave front. The denser the material the greater degree of attentuation that is experienced.

3.6.2.7 <u>End Item Tests</u>

3.6.2.7.1 Detonation Tests A and B

The primary conclusion which was derived from end item tests (Detonation Tests) A and B was that the packing materials employed in end item containers contributed significantly to the inhibiting of propagation within a container as well as container to container. This conclusion is based on the results of the five M-18 smoke grenade end item tests where each of the M-18 grenades are individually packaged in cardboard containers. These containers served to prevent propagation within the container from one item to another. The HC canisters, which are not individually packaged, showed total propagation in all A and B tests.

To provide significant data for evaluation by ASESB or the testing agency, GE-MTSD instrumented all end item tests for blast overpressure and impulse. Additionally, an optical pyrometer was utilized for flame temperature readings.

It appeared from film records and observations in HC canister tests that mass contributed significantly to the rate of reaction; i.e., there may be an exponential increase in burning rate as the mass of the sample materials increases.

3.6.2.7.2 End Item Test C (External Heat Test)

As stated in the discussion on Tests A and B, the Test C TB 700-2 specification did not require blast instrumentation or thermal measurements. However, it is felt that data which would result from this instrumentation would provide significant data relative to mass, geometric configuration, and synergistic effects.

3.6.3 PHASE I RECOMMENDATIONS

3.6.3.1 General

Based on the conclusions expressed previously, records and experience analysis, Phase I test data, and observations and evaluations by GE-MTSD and Edgewood Arsenal personnel, and the criteria under paragraph 3.6.2, certain recommendations can be made.

3.6.3.2 Detonation Test

The following recommendations are offered with respect to the TB 700-2 Detonation Test:

a. This test should be deleted as a requirement for pyrotechnics classification, since it has been demonstrated that pyrotechnics are not susceptible to detonation in the unconfined state.

b. The test procedure as applied to other materials should specify the method of containment for bulk materials, as well as requirement for consolidation of these materials if the material is consolidated as an end item.

c. A specific definition of "mushrooming of the lead cylinder" must be included in the specification. Additionally, the definition of "fragmented" must be more explicit for bulk or loose materials.

3.6.3.3 Ignition and Unconfined Burning Test

The following recommendations are offered with respect to the TB 700-2 Ignition and Unconfined Burning Test:

a. This test should be deleted as a requirement for pyrotechnics since this does not provide a definitive enough basis for determining burning rate. Additionally, the change of detonation of the pyrotechnic is extremely remote as tests have shown that these materials are not susceptible to a detonation reaction.

b. Explicit specifications should be called out for the kerosene and sawdust materials used in this test for other materials. Consideration should be given to using alcohol as the flame supporting medium.

c. As stated previously relative to the Detonation Test, confinement and configuration should be more specifically defined for bulk, loose materials.

3.6.3.4 Thermal Stability

The following recommendations are made relative to the TB 700-2 Thermal Stability Test:

a. Consideration should be given to requiring a thermocouple in the sample cube to record possible temperature deviations as a function of time. The thermocouple and recorder would also provide a means of determining the point in time and temperature when an explosion or fire occurred.

b. Consideration must be given to utilizing differential thermal analysis (DTA) and thermogravimetric analysis (TGA) for sensitivity/classification determinations of pyrotechnics. These laboratory techniques provide greater accuracy and control than the present system.

c. The definition of a "change in configuration" should be more clearly defined in TB 700-2.

d. In lieu of a DTA or TGA type test, a thermal stability test should be considered which would provide data as to what magnitude of thermal environment the material could endure without explosion, detonation, or burning; i.e., an autoignition type test would provide more meaningful, usable data than a simple "go-no-go" constant temperature test.

e. Comments made previously with regard to configuration and confinement of the sample also apply to the Thermal Stability Test.

3.6.3.5 Impact Sensitivity Test

The following recommendations are made relative to the TB 700-2 Impact Sensitivity Test:

a. The specified sample size should be increased. The existing TB 700-2 specified sample size (10 mg) precludes an assurance that a representative sample will be drawn with any significant degree of probability. For many pyrotechnic materials, a few granules of a single constituent may weigh the required 10 mgs. If the few granules are the more sensitive of the constituents, the sample material may detonate. A single detonation induced by the factors described above can cause the material to be classified Military Class 7 instead of Class 2. Increasing sample size could provide a positive statistical factor in assuring that a representative sample is selected. See paragraph 3.9 for a discussion of tests performed with varying weights and drop heights.

b. An increase in the number of samples run on each compound would provide a greater statistical probability that the reaction occurring represents to some degree the reaction that one could expect from the compound.

c. TB 700-2 should call out procedure methods and standards for blending or reblending samples to be tested, particle size requirements for the sample, and special preparation provisions for certain types and classes of materials.

d. There should be some investigation into the merits of using the Bureau of Explosives impact apparatus as an entirely different concept may be required for pyrotechnics.

e. If impact tests are to be a requirement for classification testing of pyrotechnics, some consideration should be given to testing the materials at varying weights and/or heights until a positive reaction of some kind occurs.

f. Because of the relative importance of temperature to the test environment, test equipment and materials, TB 700-2 temperature control requirements should be tightened. Additionally, conditions of humidity must also be specified in order to provide valid, reliable and accurate test data.

g. For any impact test, there must be a more clearly defined method for stabilizing the apparatus. It is very probable that the impact test results could be biased by the method that was employed to restrain or cushion the apparatus.

h. Increasing sample weight or providing instrumentation to detect the reaction should be investigated as difficulty was often experienced while running impact sensitivity tests in either hearing or seeing the reaction that occurred. This was usually true on a marginal test and might require a rerun of the sample to confirm the reaction. (See paragraph 3.3.2 through 3.3.8 for a discussion of an instrumented impact test apparatus.)

3.6.3.6 Card Gap Test

For the Card Gap Test to be effective, sympathetic detonation must occur in the acceptor material, but pyrotechnics have shown no indication of this. Therefore, because the Card Gap Test does not provide a valid means of classifying or measuring the sensitivity of a pyrotechnic material, it is recommended with respect to the Card Gap Test as specified by TB 700-2 that, for materials that could meet the sympathetic detonation criteria, the Card Gap Test procedure be more clearly defined with respect to: (1) witness plate materials - too hard or brittle a plate could bias the test by shattering rather than having a hole punched in the plate; (2) witness plate stand configuration - the stand is specified as being required to support the plate on two edges, whereas the picture in the specification (TB 700-2) shows a stand which supports the plate at four corners.

3.6.3.7 End Item Tests

The following recommendations are made with respect to the End Item Tests A, B, and C in TB 700-2:

a. The test procedure should require additional instrumentation to the extent that blast overpressure and impulse can be recorded for all pyrotechnics end item tests.

b. The procedure should also require instrumentation for recording of temperatures during all of the pyrotechnic end item tests.

c. To record the significant test events such as explosion and subsequent fragment dispersion, it would be judicious to require color motion picture coverage for end item tests. Camera speeds in the neighborhood of 500-3000 frames per second are recommended for this application.

d. Although it may be beyond the scope of TB 700-2 testing, consideration must be given to packaging and packaging methods employed for pyrotechnic end items. The results of the end item tests discussed previously indicate that flame attentuation is possible for pyrotechnics.

3.7 SEGMENT 2 - GENERAL OBSERVATIONS

The tests reviewed in this section excluding the TB 700-2 tests were generally inconclusive within themselves. The samples tested were unfortunately in the same "family"; i.e., they did not exhibit radically different reactions with the exception of the fuel mix and $KClO_3$ S mixture. In order to establish a reaction/result versus hazard potential scale it is required that (1) a wider divergence of sample materials be examined; (2) many more tests of this type be conducted; and (3) a sophisticated data evaluation/correlation system be established. The latter requirement is probably the most important, in that much data is available, not only from this program but from many other sources; e.g., acceptance testing, field experience, manufacturing data and experience, quality control records, and many other commercial as well as military sources. The parametric relationships of this chemical, environmental, and physical data, when evaluated in terms of the pyrotechnic environment, will provide the basis for further hazards criteria.

It has been shown that physical and chemical properties, as determined by DTA, Parr, electrostatics, and other means, can be related to hazards. If properly defined and statistically verified, these values can be correlated to provide a hazards scale. In Section 5, an attempt is made to correlate and compare Phase I and III data in order to determine if there is a possible rank or degree of hazard within these closely related compositions. Table 5-1 shows some indication that Class 7 compounds and lactose compounds have a higher "hazards rating." This may be a beginning of a damage/hazard index.

SECTION 4
SEGMENT 3 - DEVELOPMENT OF NEW AND/OR MODIFICATION OF EXISTING EQUIPMENT AND/OR TEST METHODS

4.1 INTRODUCTION

The objectives of this segment of work, as stated in the Contract Scope-of-Work, were as follows:

a. Perform an evaluation of existing tests and equipment and the ability of these tests and equipment to measure the pyrotechnics' sensitivity to certain stimuli. Typical examples of the stimuli are as follows:

- Electrostatic
- Friction (friction shoe, swinging pendulum)
- Mechanical Impact (Bureau of Mines, Bureau of Explosives, Picatinny Arsenal)
- Thermal (international heat test, DTA, closed bomb)
- Detonation (card gap, standard detonation, TNT Equivalency)

b. Using a Card Gap as an example, the activities anticipated in the segment would be as follows:

- Modification of donor/acceptor parameters, i.e., materials relative sizes, configuration.
- Design and fabrication of prototype equipment required to obtain test data necessary for the classification of pyrotechnics.
- Development of procedures for use of above equipment.
- Testing to validate adequacy of equipment and procedures.

Under part (a) above, electrostatics testing and mechanical impact have been evaluated under Section 3. Friction stimuli were not investigated in Phase III primarily because it was felt that the majority of these tests rely on the same physical and chemical laws as the impact test; e.g., creation of a hot spot in the material which communicates to other material. The specified resultant reaction for these tests is usually the same as impact; i.e., decomposition, explosion, smoke, odor, etc., which require the traditional SSS (sound, sight, smell) evaluation. The anomalies associated with friction sensitivity tests would be the same as those for the impact sensitivity tests discussed in Section 3 above.

Evaluation of other "existing tests and equipment" are reported in paragraph 4.2. These are the tests which were performed in conjunction with Phase I. They were performed at that time to take advantage of the on-going test program. This provided data from identical sample batches, similar test hardware, instrumentation, and equipment. It was, in other words, an effort to minimize costs and test variables.

Part (b) requirements are satisfied by paragraphs 4.3, 4.4, 4.5, and 4.6 following. The tests described therein are Hartmann dust reaction tests, HE equivalency, spark impingement tests, and instrumented Parr Bomb. Each section contains specific conclusions and recommendations; however, Section 5 contains a summary of the overall conclusions and recommendations.

4.2 EVALUATION OF TB 700-2 TESTS

4.2.1 GENERAL

Anomalies associated with TB 700-2 tests are reported in Section 3 preceding. Following is a report on tests which were conducted to investigate the effects of changes to the basic TB 700-2 test setups, configuration, instrumentation, and procedures, which might provide an insight into the basic causes of the anomalies.

The following is a list of the changes and recorded data discussed in the following paragraphs:

- Detonation Test
  - Container for Bulk Material
  - Importance of Initiator Placement
  - "Go-No-Go" Gage and Application
- Thermal Stability - Additional Instrumentation
- Card Gap
  - Pyrotechnic Contribution
  - Card Gap Configuration (Horizontal, Inverted, Normal)
  - Special Witness Plates
  - Witness Plate Deformation
- Ignition and Unconfined Burning - Change in Configuration
- TNT Equivalency
  - Deviation from Trauzl Block Test
  - Basic Premise
  - Comparison
  - Results
  - Test Method

- High Speed Motion Picture Photography
  - Fragment Dispersal
  - Fragment Velocity
  - Fireball Growth

4.2.2 DETONATION TEST

All 2-inch cube samples for the detonation test were fabricated from Whatman No. 4 ashless filter paper. This paper is sufficient to contain the sample and hold a cube configuration. Low residue and a moderate burning rate make filter paper an acceptable container material. It is felt that a material such as this should be specified in TB 700-2 for loose, granular or bulk material.

In an attempt to examine the detonation test in greater detail, two special tests were run. In the first of these tests, the No. 8 blasting cap was inserted into the 2-inch cube sample as far as possible, i.e., until the blasting cap was separated from the lead block only by the thickness of filter paper used to form the 2-inch cube sample container.Figures 4-1 and 4-2 show the end of the lead cylinders used in each of two tests. The photographs show an indentation in the lead due entirely to the vertically directed explosion of the No. 8 cap. In each test, the sample material scattered without burning and slight deformation of the lead cyclinder occurred. In another special detonation test the No. 8 blasting cap was positioned 2 inches above the surface of the 2-inch cube sample. There was no measurable difference in the configuration of the lead cylinder after two such tests were conducted on the same cylinder (see Figure 4-3). The conclusion drawn was that the placement of the initiator makes a measurable difference in the distortion of the lead cylinder and care should be taken to specify in TB 700-2 as to exact placement of the initiator.

In an effort to develop a quick and simple means of checking for deformation of the lead cylinder, a "go-no-go" gage was fabricated. This device is shown in Figure 4-4. It is simply placed over the cylinder, and run up and down the entire length, and a determination made that the cylinder does or does not exceed the original 1-1/2 inch diameter dimension by more than 1/16 inch at any point along its vertical axis. The purpose of the "go-no-go" gage is to standardize the deformation definition as prescribed in TB 700-2.

4.2.3 THERMAL STABILITY TEST

In all thermal stability tests conducted on pyrotechnic samples, the 2-inch cube sample was placed in the ventilated explosion-proof oven with a copper-constantan thermocouple, in addition to the instrumentation required by TB 700-2, inserted in the sample material. The purpose of this thermocouple is to record any exothermic or endothermic reactions of the pyrotechnic composition under test.

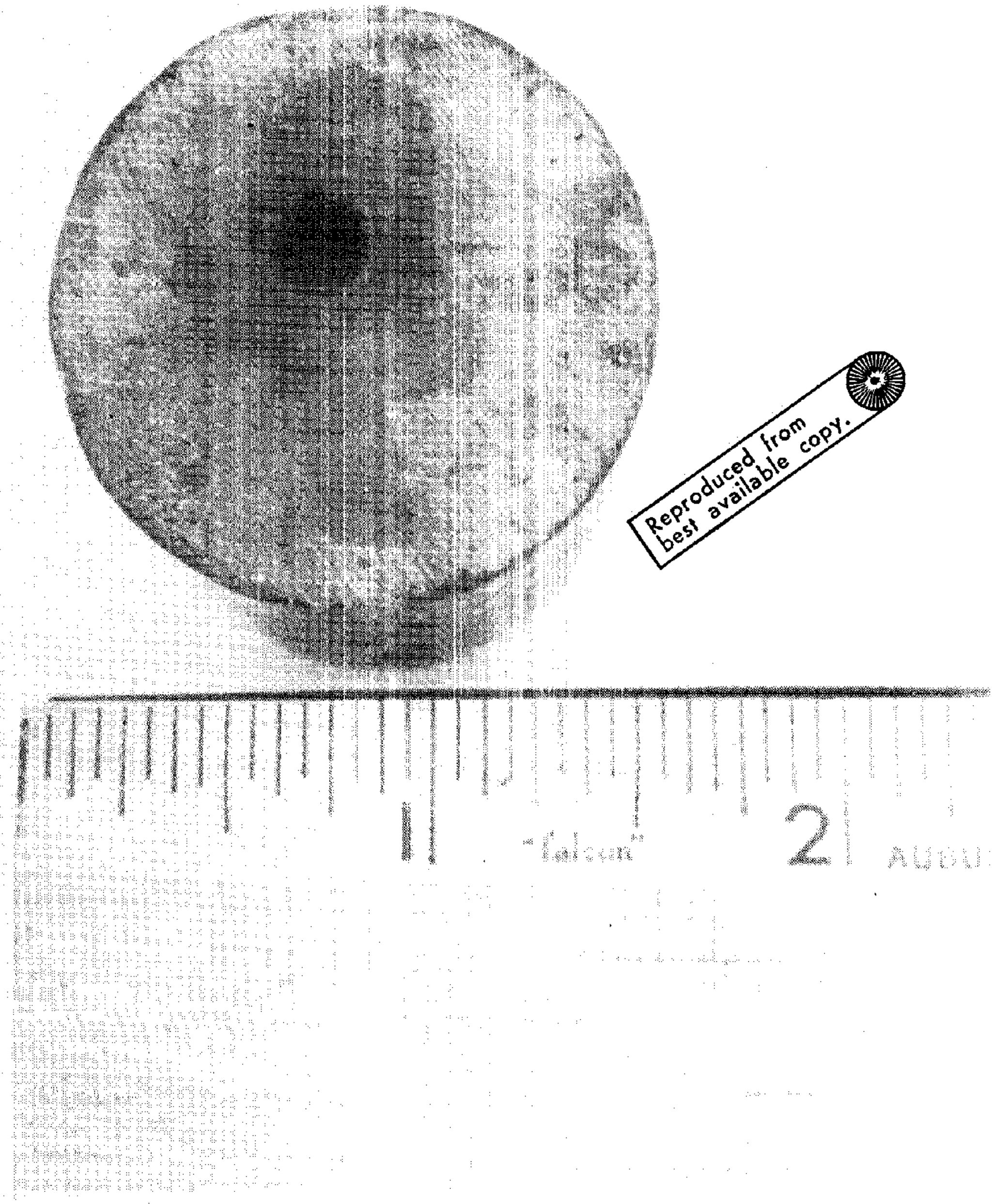


Figure 4-1. Special Detonation Test (#1 - Cap in Sample)

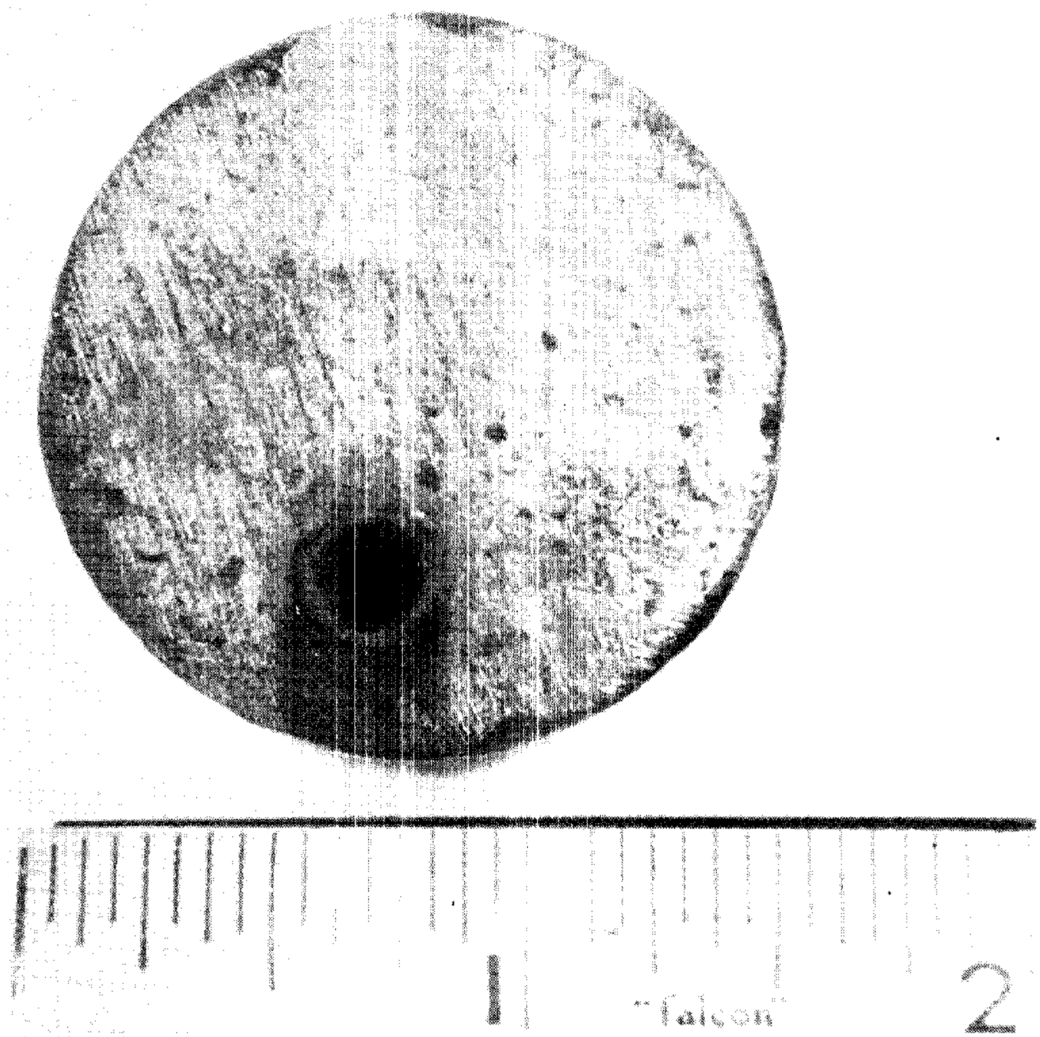


Figure 4-2. Special Detonation Test (#2 - Cap in Sample)

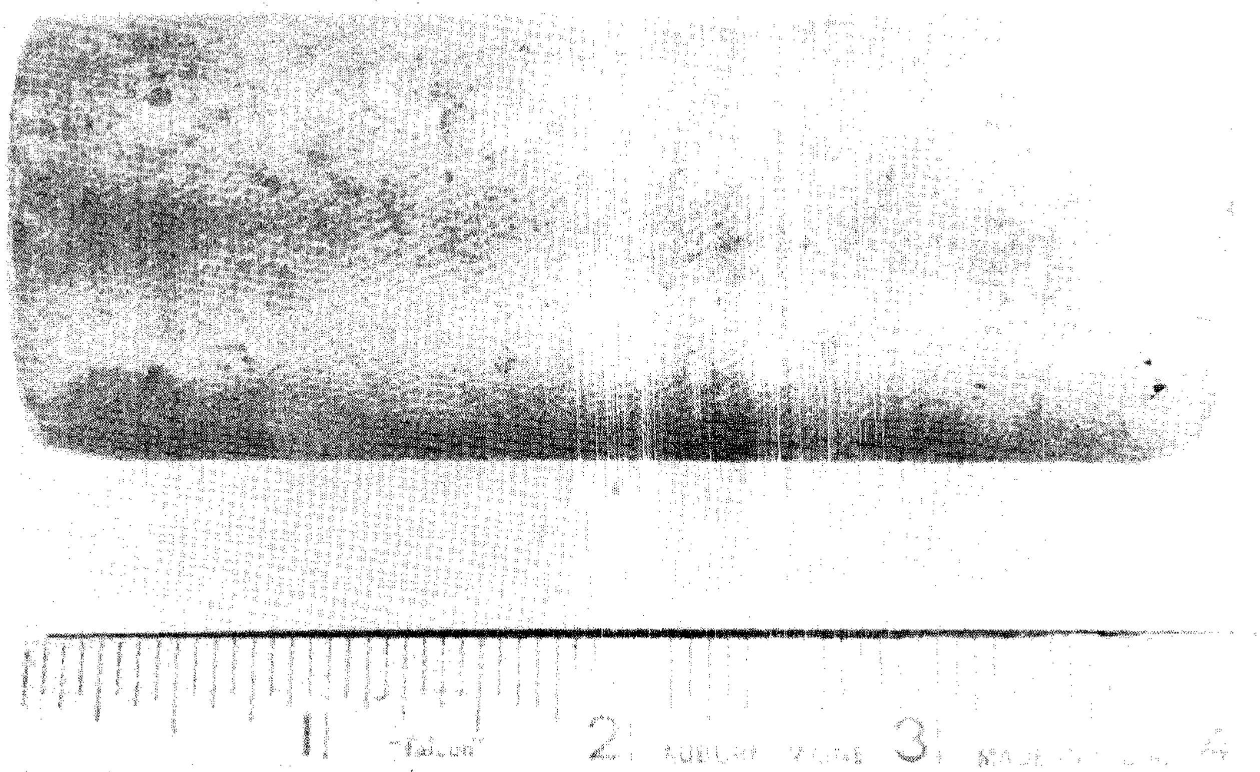


Figure 4-3. Special Detonation Test (#3 - Cap 2" above Sample)

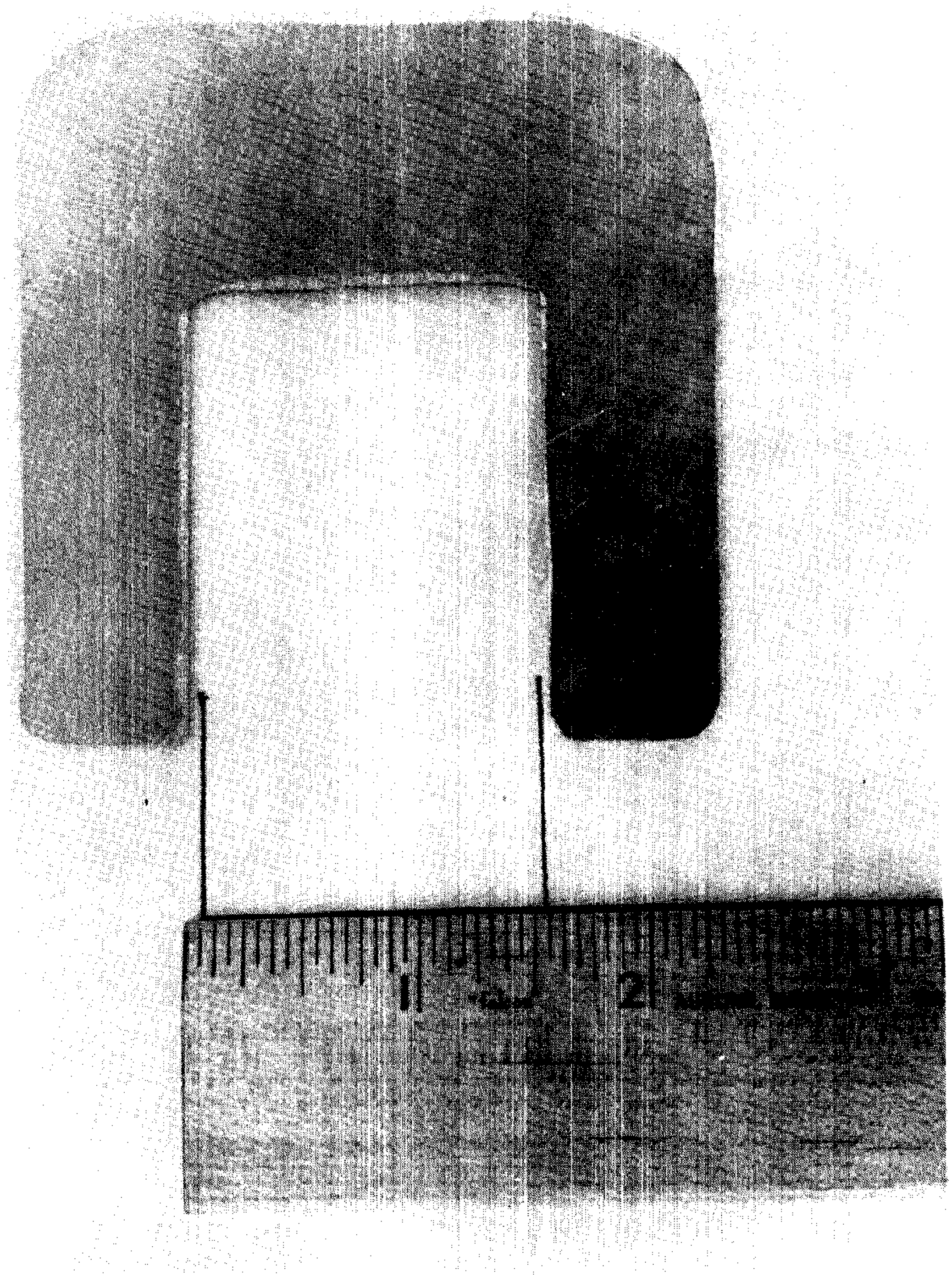


Figure 4-4 Detonation Test - Go-No-Go Gage

4.2.4 CARD GAP

4.2.4.1 Pyrotechnic Contributions

To determine the contribution of the pyrotechnic material under test, the following three types of tests were performed to determine if the pyrotechnic material was a contributing factor:

- Empty Tube
- Inert Filled Tube
- Instrumented Card Gap

4.2.4.2 Empty Tube

A test was performed using an empty 5-1/2 inch sample pipe with all other card gap hardware and configuration remaining unchanged. The results of this test is shown in Figures 4-5 and 4-6. Examination of the plate indicated that the deformation (2-5/8 inches) was more extreme than any of the eleven pyrotechnic samples.

4.2.4.3 Inert Filled Tubes

A tube was filled with coarse washed sand. The results of this test is shown in Figures 4-7 and 4-8. Examination of the witness plate showed little or no deformation.

4.2.4.4 Instrumented Card Gap

A standard card gap test was performed on the eleven pyrotechnic samples. The results of these tests are shown in tabular form in Table 4-1. Examination of the data indicated that there was some contribution on certain compounds, such as CS, HC, FM mixes whereas, in the other tests little or no contribution was recorded.

The conclusion drawn from the three methods mentioned above, was that the instrumented card gap tests of the eleven pyrotechnic compositions indicated that there was no detonation of the material and that these samples under test actually attenuated the pentolite booster charge.

4.2.4.5 Card Gap Configuration

During performance of the instrumented card gap tests it was desirable to determine the optimum configuration for maximum data acquisition by the pressure transducers. An inert material was tested in three configurations as follows:

- Inverted - 180° from normal
- Horizontal - 90° from normal
- The normal configuration per TB 700-2

Figure 4-5. Card Gap Witness Plate, Sample Pipe Empty

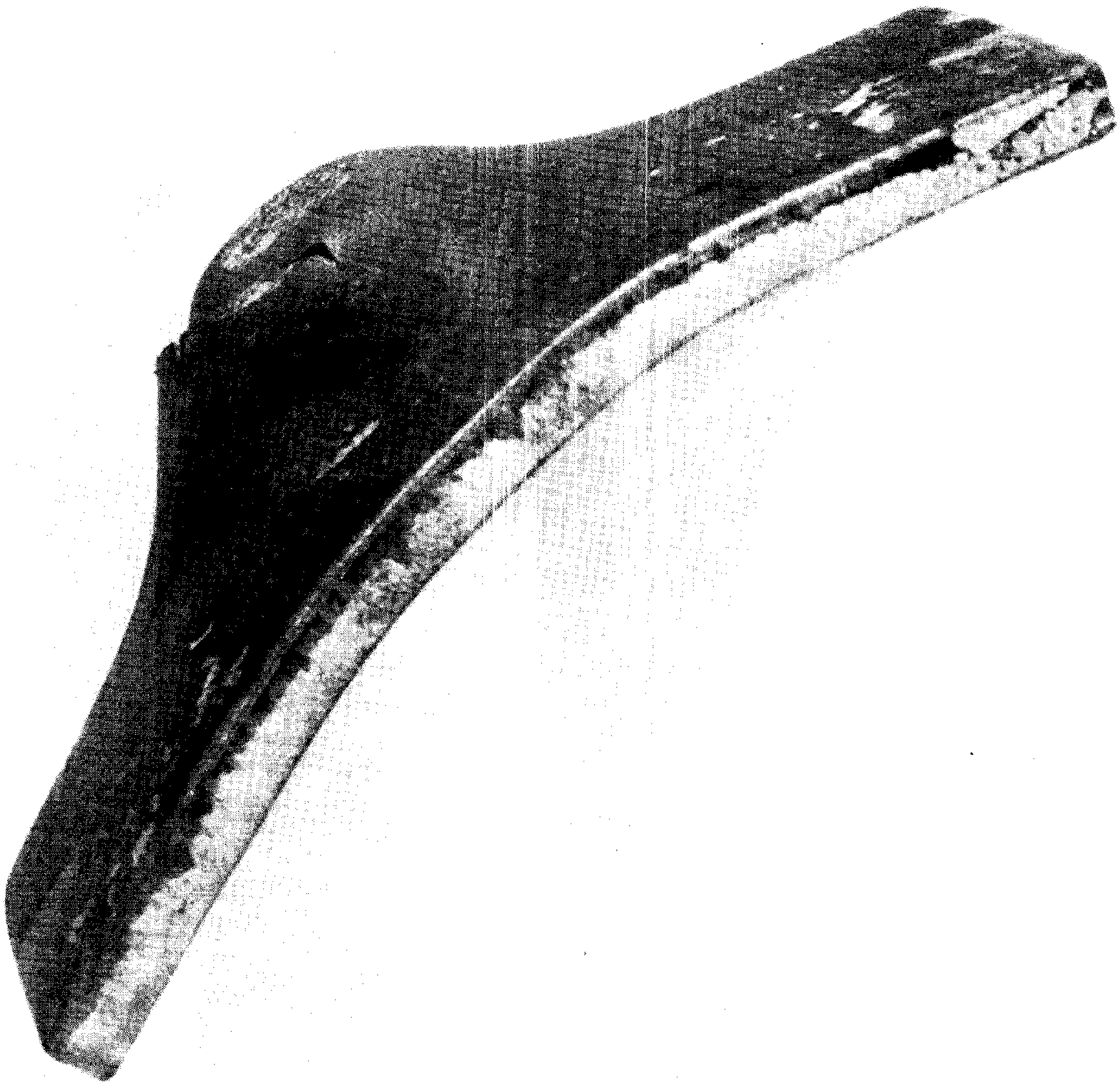


Figure 4-6. Card Gap Witness Plate, Sample Pipe Empty

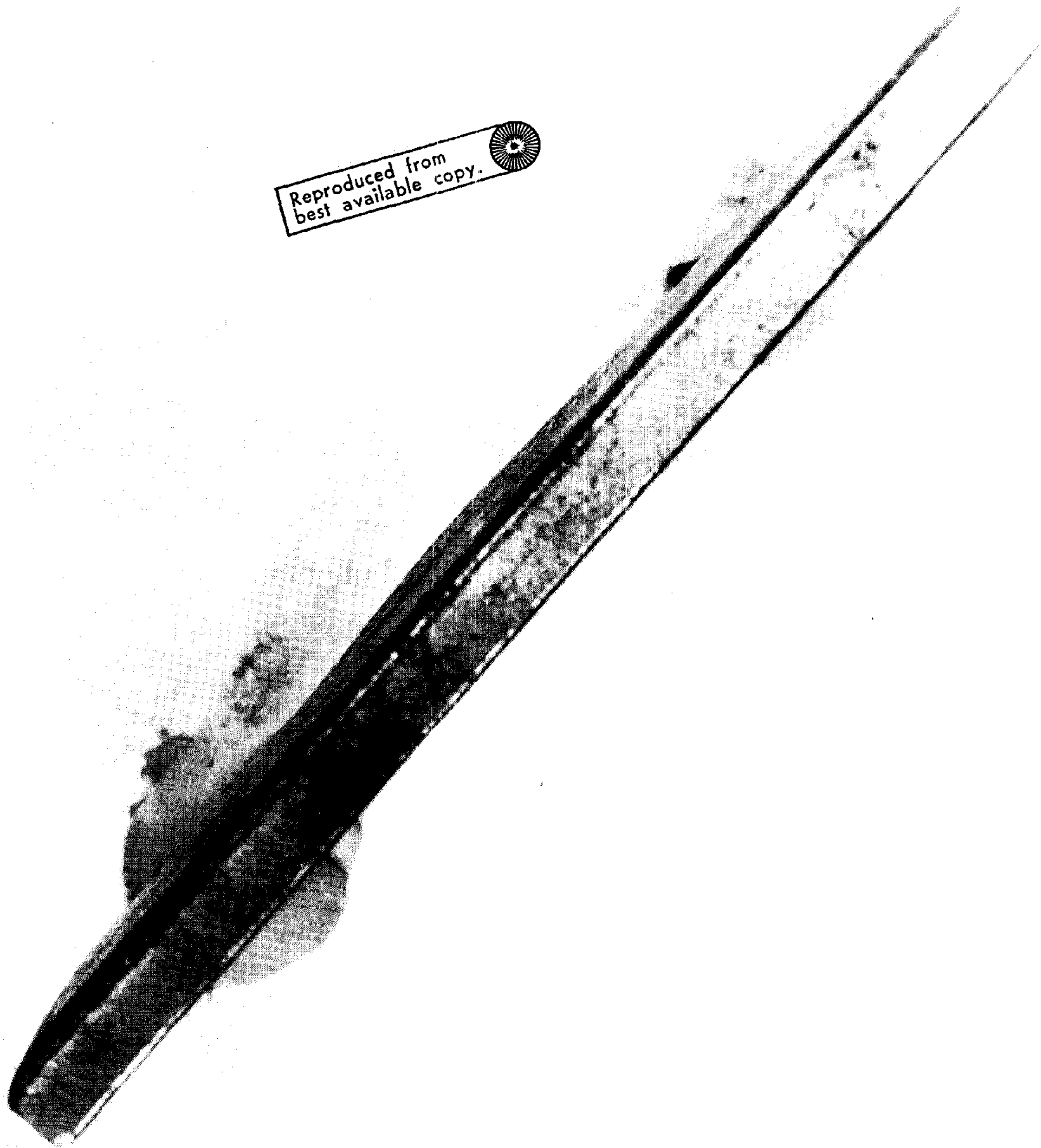


Figure 4-7. Card Gap Witness Plate, Sand-filled Pipe

Figure 4-8. Card Gap Witness Plate, Sand-filled Pipe

Table 4-1. Instrumented Card Gap Data

SAMPLE MATERIAL	MEAN PEAK OVERPRESSURE (PSI)	*CALCULATED SCALED DISTANCE (Z) ($R/w^{1/3}$)	**PEAK OVERPRESSURE EXPECTED VALUE (PSI)	MEAN IMPULSE (PSI-MSEC)	*CALCULATED SCALED DISTANCE (Z) ($R/w^{1/3}$)	**IMPULSE EXPECTED VALUE (PSI-MSEC)
SG 3-69-1	35.37	4.91	30.05	11.67	4.11	9.759
SR 3-69-1	32.00	5.11	30.05	10.38	4.84	9.759
SY 3-69-1	31.50	5.15	30.05	10.42	4.81	9.759
SV 3-69-1	30.75	5.20	30.05	10.93	4.51	9.759
LG 3-69-1	32.87	5.06	30.05	10.49	4.77	9.759
LR 3-69-1	30.62	5.21	30.05	10.43	4.81	9.759
LY 3-69-1	33.87	4.99	30.05	11.16	4.38	9.759
LV 3-69-1	34.00	4.98	30.05	11.38	4.26	9.759
FM 3-69-1	33.12	5.04	30.05	11.01	4.46	9.759
HC 3-69-1	33.37	5.02	30.05	9.55	5.40	9.759
CS T-752	35.35	4.91	30.05	10.97	4.49	9.759

*All charges were fired at a "Z" value of 5.25. The calculated "Z" value is based on the mean actual overpressure or impulse recorded from the test.

**The expected values for peak overpressure and impulse are based on 161 grams (w) of pentolite at a distance of 3.717 feet (R).

The result of these tests indicated that for optimum instrumentation measurement, the normal configuration (as specified in TB 700-2) provides more consistent data than the other two methods tested.

4.2.4.6 Witness Plate Deformation

Correlation of witness plate deformation with the overpressure and impulse data from the instrumented card gap was attempted.

The data evaluated for correlation included the following:

- Depth of Deformation
- Volume of Deformation
- TNT Equivalency Value Based on Impulse
- TNT Equivalency Value Based on Blast Overpressure

The value for each of the bulk compounds tested and the corresponding data accumulated from the instrumented card gap information is shown in Table 4-2.

Table 4-2. Witness Plate Deformation

TEST MATERIAL	CARD GAP DATA % TNT BASED ON OVER PRESSURE (PSI) CLOSED TUBE	 % TNT BASED ON IMPULSE (PSI-SEC.) CLOSED TUBE	AVERAGE DEFORMATION DEPTH (INCHES)	AVERAGE VOLUME OF DEFORMATION (CC.)
SG 3-69-1	4.30	0.57	1-7/16	71.1
SY 3-69-1	3.72	0.92	1-5/16	97.2
SR 3-69-1	5.35	1.11	1-3/8	74.1
SV 3-69-1	6.53	1.26	1-15/32	71.6
LG 3-69-1	6.31	0.99	1-1/2	75.3
LY 3-69-1	7.11	1.42	1-13/16	96.6
LR 3-69-1	5.40	0.62	1-9/16	-
LV 3-69-1	4.72	0.68	1-11/16	91.3
FM 3-69-1	10.88	3.32	1-23/32	101.9
HC 3-69-1	-	-	7/8	-
C/S T-752	10.36	2.94	1-3/16	-

4.2.4.7 Special Witness Plate

Edgewood Arsenal was experiencing a test anomally relative to their witness plate tests. Their plates were shattering rather than undergoing deformation or penetration. Therefore, GE-MTSD, at the request of Edgewood, ran a series of tests comparing several witness plates being used by Edgewood with several of those witness plates being used by GE-MTSD. All other variables were held constant.

The results of the tests established that the witness plates supplied by Edgewood shattered and the witness plates supplied by GE-MTSD did not shatter under the same test conditions. A physical and chemical analysis was performed with the following results:

- Witness plate supplied by Edgewood - Type of Steel 1010
Hardness 87.1
Rockwell "C"
Tensile Strength 105,000 psi
- Witness plate supplied by GE-MTSD - Type of Steel 1010
Hardness 58
Rockwell "B"
Tensile Strength 62,200 psi

It can be seen from the above data that the Edgewood plate is harder and therefore much more brittle, thus it has the tendency to shatter in a higher precentage of tests. Had the Edgewood or GE-MTSD plates been "softer" to the same relative degree, the card gap test results may have been substantially altered. In other words, it may have been possible to punch a hole in a "softer" plate.

4.2.4.8 Volumetric Measurement

To correlate data between sample size, weight of charge, and detonation effects of card gap test, volumetric measurement of the depression in the witness plate was attempted.

It was determined that filling the depression with a measured quantity of water provided the greatest degree of accuracy. By leveling the plate and allowing the meniscus of the water to touch the level, more reproducible results could be attained. Each witness plate was measured four times to account for disparity in the deformation. Figure 4-9 shows the volumetric measurement test setup.

4.2.4.9 Depth of Deformation

Examination of the 6-inch square steel witness plates revealed only slight differences in configuration of the plate. Deformation ranged from 7/8 inches for HC smoke mix to 1-13/16 inches for Lactose Yellow smoke mix. Table 4-2 shows the extent of deformation of the witness plate for the various test materials. To obtain these deformation measurements, the witness plates were sectioned and placed under a gridded glass plate. The subsequent photographs (see the typical example in Figure 4-10) served as basis for the measurements.

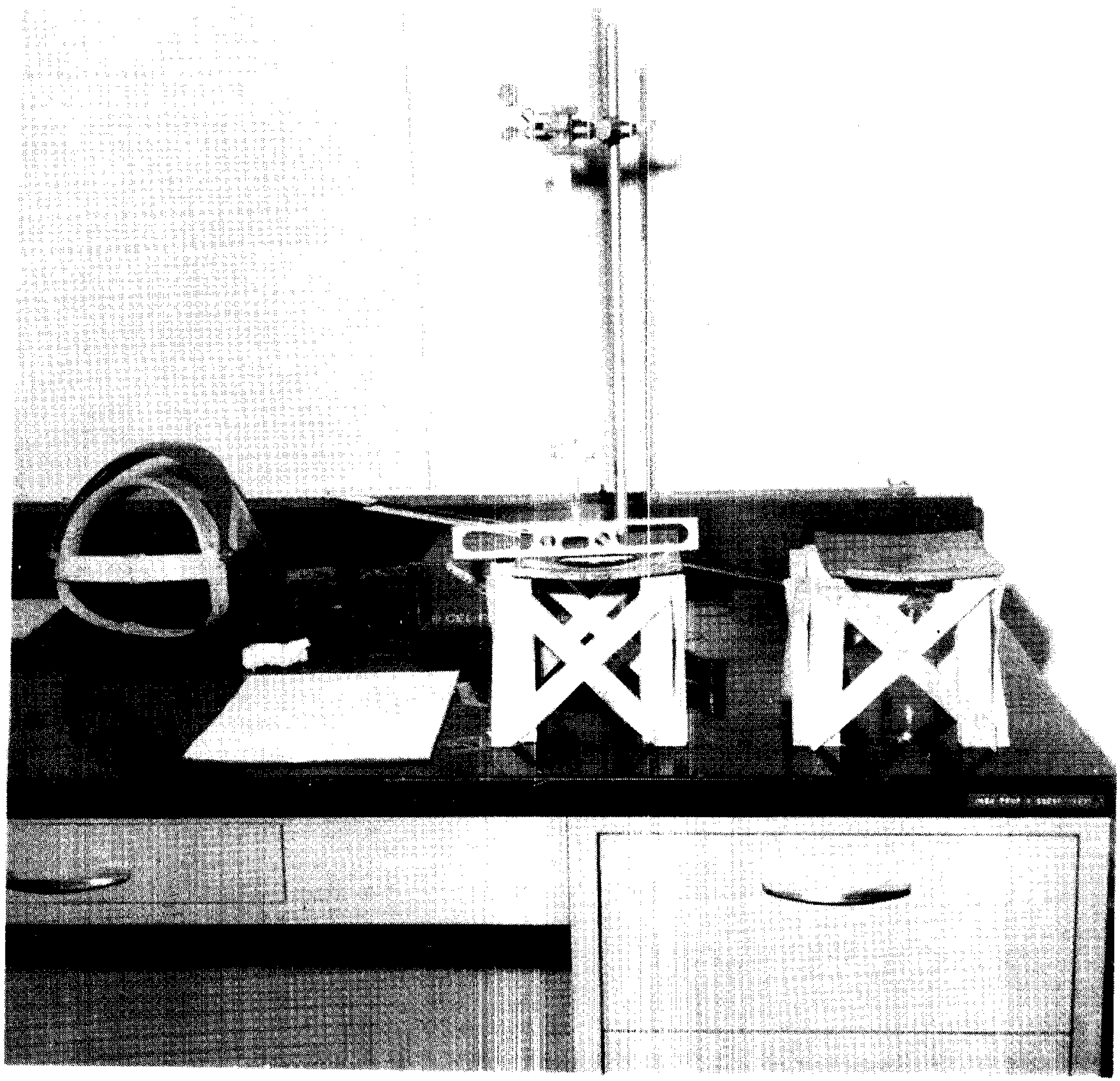


Figure 4-9. Volumetric Measurement Test Setup

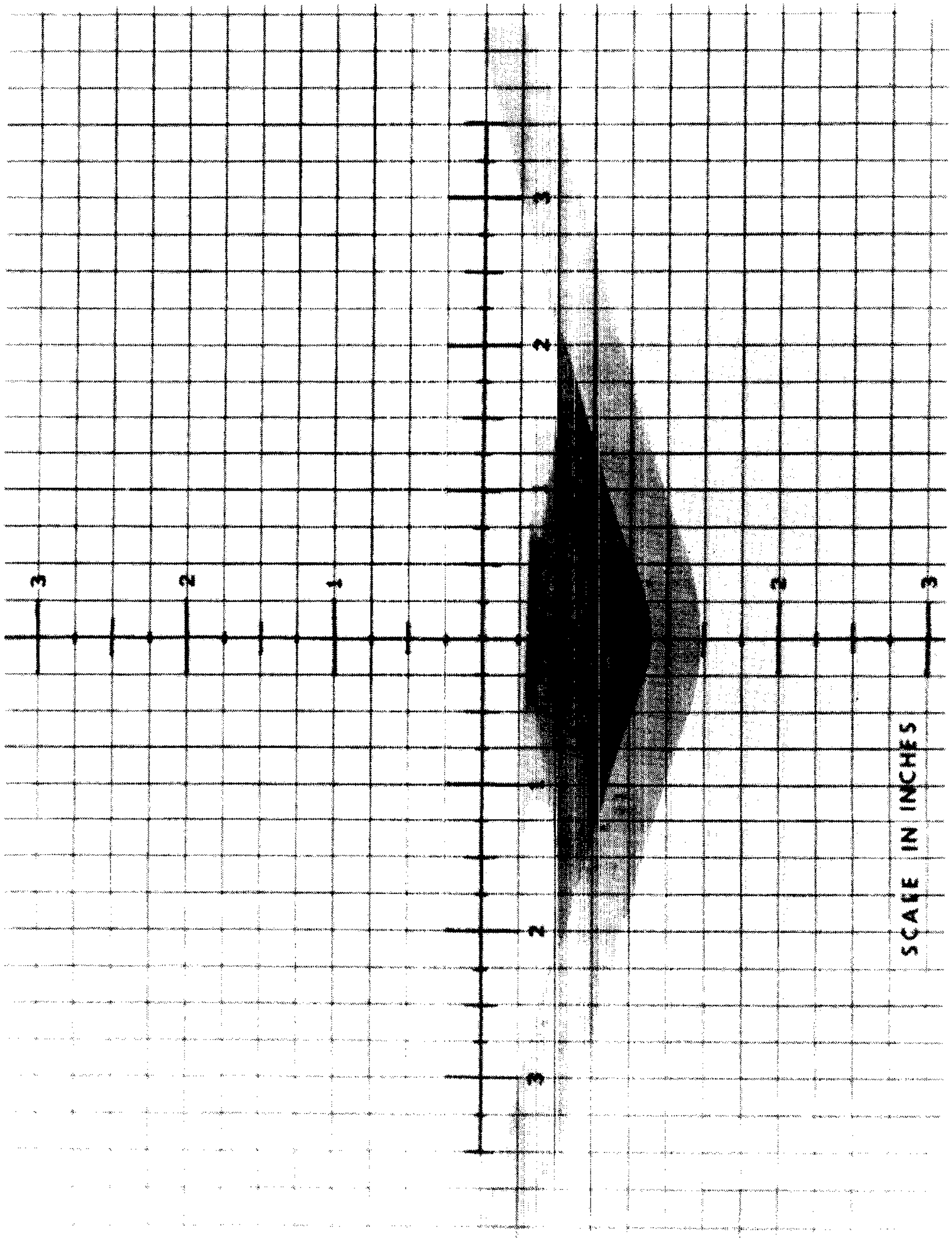


Figure 4-10. Typical Witness Plate Showing Depth of Deformation

4.2.5 IGNITION AND UNCONFINED BURNING TEST

The normal test configuration, previously discussed, was modified to examine the use of other flammable materials in lieu of the kerosene and sawdust. This effort was performed to determine if the dark smoke cloud which enveloped the sample cloud could be reduced. A prepared sample was placed in the metal tray which was then filled with alcohol. Ignition and subsequent burning of the sample and alcohol resulted in no appreciable change in either the burning time or the cloud formation. Both single cube and multiple cube tests were performed with the alcohol substitution. It was concluded that the black smoke cloud was the result of the dye decomposing.

4.2.6 TNT EQUIVALENCY

The proposed test plan required that Trauzl Block tests be performed on the eleven granular bulk samples if it was proven that they detonated by any of the prescribed tests per TB 700-2. A basic requirement for the Trauzl Block test is that the material detonate in response to a blasting cap. Since the pyrotechnic samples did not detonate in any of the TB 700-2 tests, Trauzl Block tests were not performed. In lieu of the Trauzl Block a method was derived for performance of TNT equivalency testing in a configuration which completely confined the sample.

The TNT (or HE) equivalency test program is presented in detail in paragraph 4.4.

4.2.7 HIGH SPEED MOTION PICTURE PHOTOGRAPHY

4.2.7.1 Fragment Velocity

Based on preliminary information assembled from card gap movie films taken at 50 frames per second, it was determined that for two fragments of unknown size there were velocities exhibited of between 100.0 - 293.3 feet per second. This calculation was made for the sulphur green (SG 3-69-1) bulk granular sample.

4.2.7.2 Fireball Growth

Examination of the first few frames after ignition of the card gap sample enabled a rough calculation of the fireball growth rate to be made. For those films examined, the following growth rates were determined (Table 4-3). A sample exposure sequence is shown in Figure 4-11.

Table 4-3. Fireball Growth Rate Data

SAMPLE	FILM SPEED	FRAMES	ESTIMATED RATE
SG 3-69-1	9,800 fps	2-5	4.88×10^3 ft./sec.
Inert Material	7,200 fps	2-5	4.82×10^3 ft./sec.

From this data it was concluded that the pyrotechnic sample material did not contrubute significantly to the detonation reaction. If there was an effect, it was to reduce the fireball growth rate.

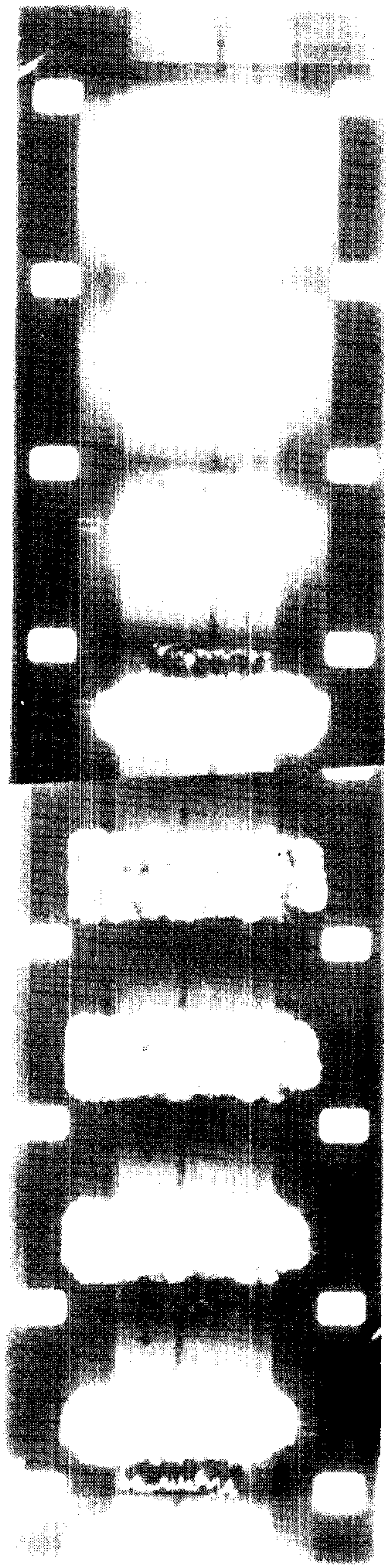


Figure 4-11. Fireball Growth Sequence @ 9800 Frames Per Second

4.2.7.3 BRL Ultra High Speed Motion Pictures

A sample of sulfur green smoke composition and card gap hardware were sent to the US Army Ballistics Research Laboratory (BRL) High Speed Photographic Laboratory for the purpose of photographing the card gap test event with the rotating prism framing camera. Motion pictures were taken of the card gap test at 500,000 fps using (a) an empty sample pipe, (b) a pipe 1/4 full and (c) a completely filled pipe. The filmed sequence was animated in order to show the event at viewable speeds. The three tests showed:

(a) Empty Pipe - A detonation wave was observed moving uniformly through the sample pipe without the pipe rupturing. This confirms the actual test results discussed above.

(b) 1/4 Sample in Pipe - The sample material was observed being pushed through the tube with no apparent initiation taking place within the material.

(c) Full Sample in Pipe - In this test, the sample material was again being shoved through the pipe without ignition taking place. Initial rupturing of the pipe into the characteristic "banana peel" strips was also observed in this film series.

4.3 HARTMANN DUST EXPLOSION TESTS

4.3.1 RATIONALE

Evaluation of the hazards associated with the manufacture of pyrotechnics (i.e., pressing, mixing, sifting, screening, filling, etc.) requires consideration of the environment. An important element of the environment associated with pyrotechnic processing is the concentration of the various ingredients as settled or suspended dust in the immediate workfloor area.

In any hazard appraisal, it is important that the ignition sensivitity of the pyrotechnic materials in suspension in air to the potential stimuli available be explored in detail. Along these lines, testing should be designed to determine whether the following concepts might be applied in the manufacture of pyrotechnics in order to reduce the safety hazards of operations which involve the emission of dust clouds as a by-product:

- Evaluation of sensitivity of the various ingredients and substitution by less sensitive materials where possible without changing performance characteristics.
- Minimization of the percent of material passing through the smaller sieve sizes without degrading functional characteristics. It may be that some ingredients will burn as well in larger particle sizes.
- Use of inert fluids (that tend to consolidate materials) to aid in preventing dust cloud formation.

- Determination of the sensitivity of pyrotechnic dusts to the various stimuli available, specifically, if one particular ignition source; e.g., open flames, glowing particles, heated surfaces, electric arcs, static discharge, frictional sparks, is more effective than another.

This determination should be made in view of the fact that the size, duration, and intensity of the ignition source affect the lower explosive limit of the dust cloud.

4.3.2 TECHNICAL APPROACH

4.3.2.1 Description

In order to evaluate pyrotechnic dust hazard characteristics, experimental work on explosibility of pyrotechnic dusts was performed in a special laboratory scale apparatus (Figures 4-12 and 4-13) developed by the Bureau of Mines.

Tests were designed to evaluate the ignition threshold of pyrotechnic dust atmosphere by determining:

- Minimum electrical energy of ignition as a function of dust particle density, humidity, and stoichiometric ratio.
- Maximum reaction induced pressure and rate of pressure rise as a function of chemical imbalance of stoichiometric ratio.

Basically, the chamber is a 2-3/4 inch diameter steel tube, 12 inches long that is vertically mounted. Shown in Figure 4-14 is the support for the steel chamber. The interior of the stand consists of the following:

- Dispersion Cup - (where weighted sample is placed).
- Adjustable compressed air deflector - (in order to deflect compressed air onto sample).

The sequency of operations for the pneumatic regulator system is as follows: (See Figure 4-15)

- Compressed air was supplied by a "K" bottle on top of which was mounted a manually operated gate valve - all bottles were certified by GE Quality Control to be "missle grade" air (particulate material less than 50 microns and Dew Point at 75°F).
- A manually operated block valve was connected through a flex line to the "K" bottle in order to isolate the "K" bottle from the system, thereby providing greater safety for the operator.
- A regulator valve rated at 1000-psi capacity (with ball-type vent valve) was connected downstream of the block valve. This valve was used to regulate the pressure applied to the accumulator.

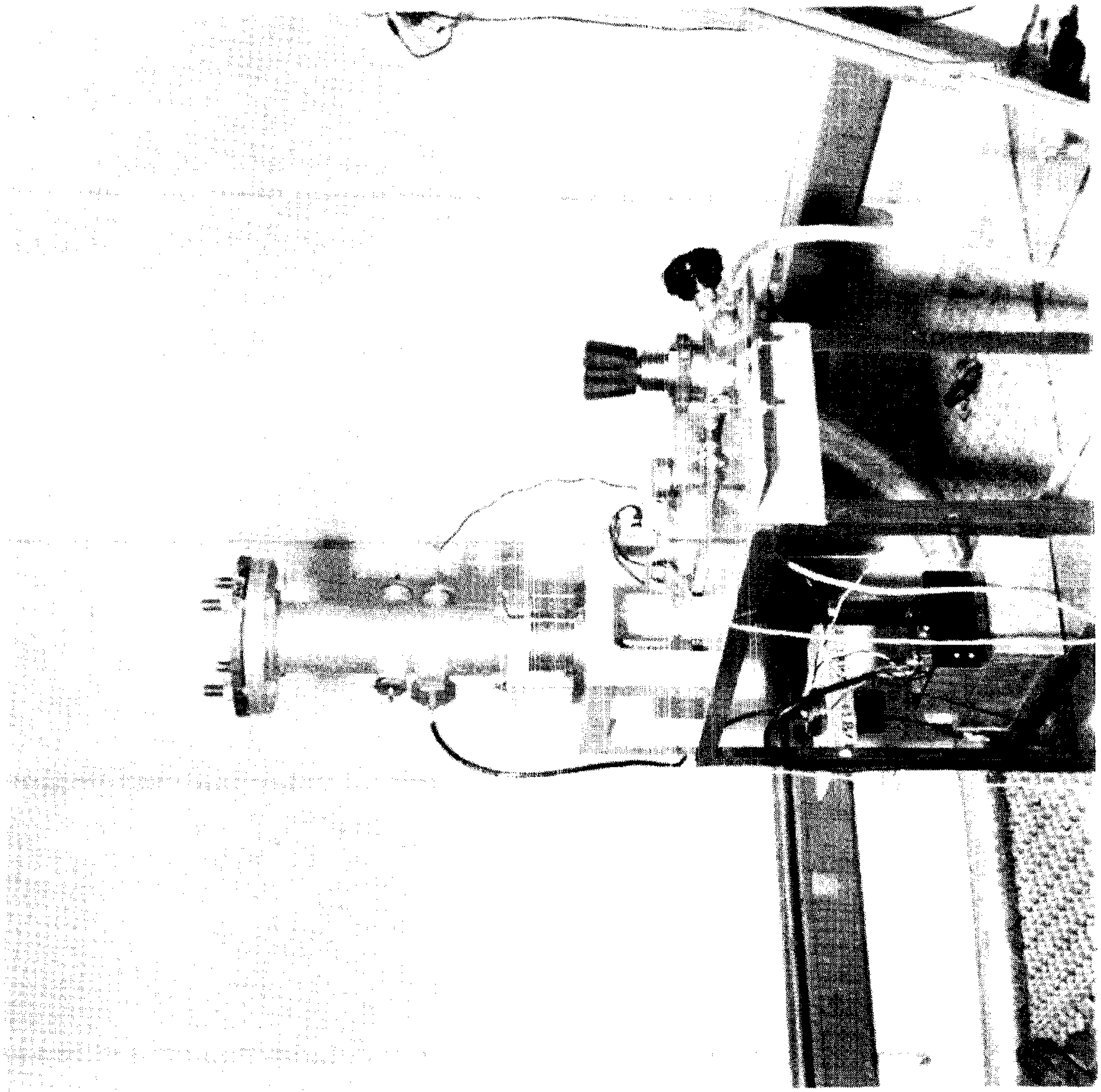


Figure 4-12. Bureau of Mines Hartmann Apparatus

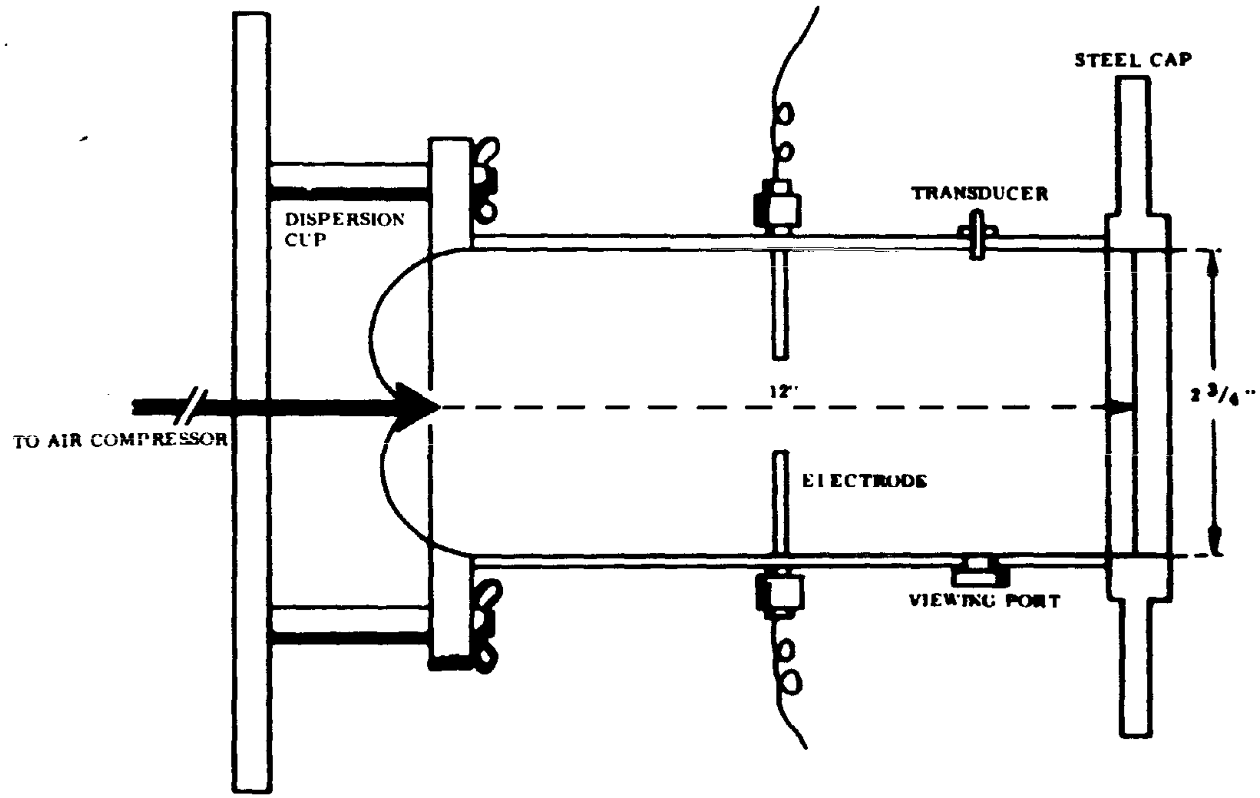


Figure 4-13. Cutaway of Hartmann Chamber

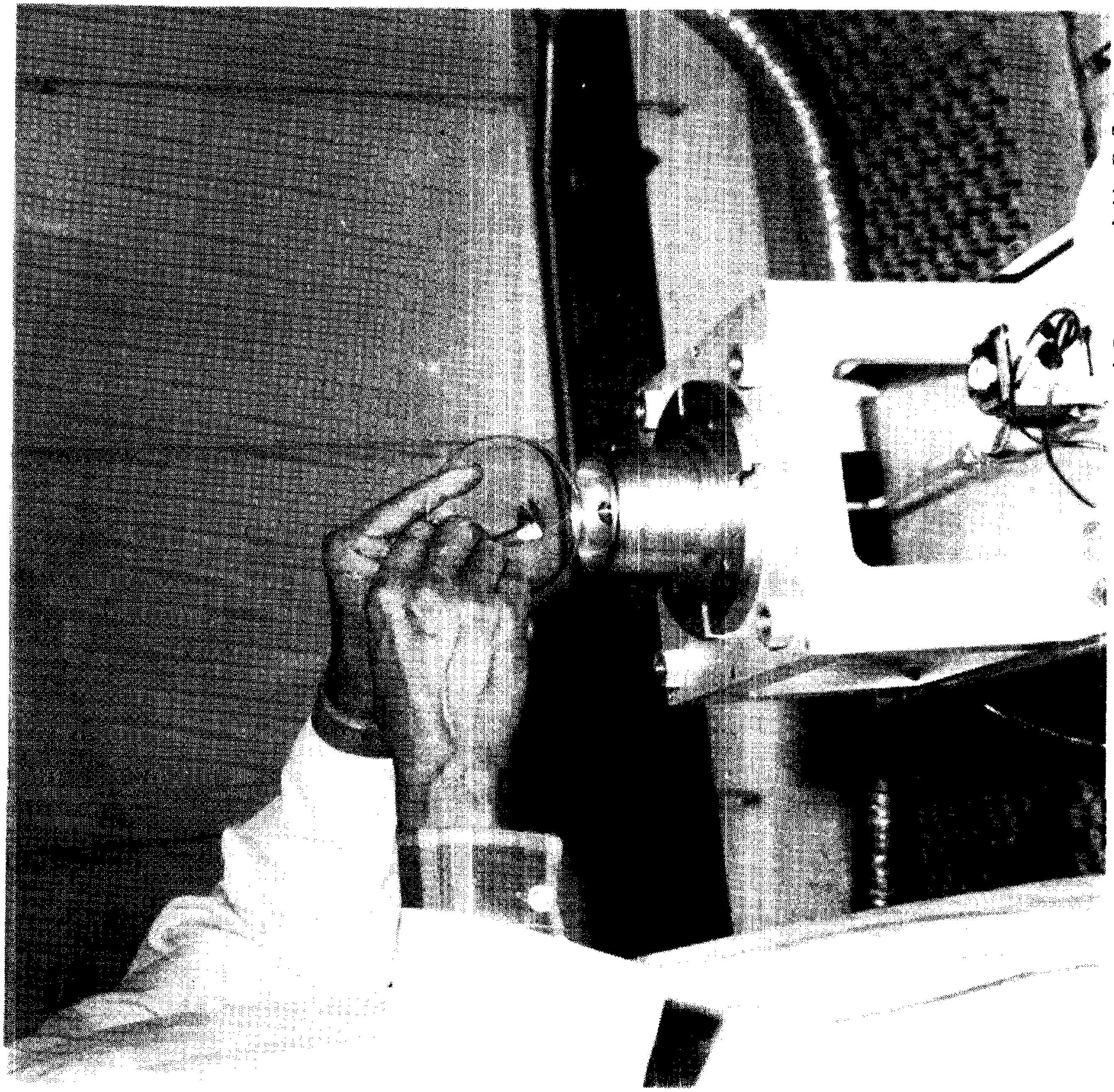


Figure 4-14. Support Stand Showing Dispersion Cup and Compressed Air Deflector

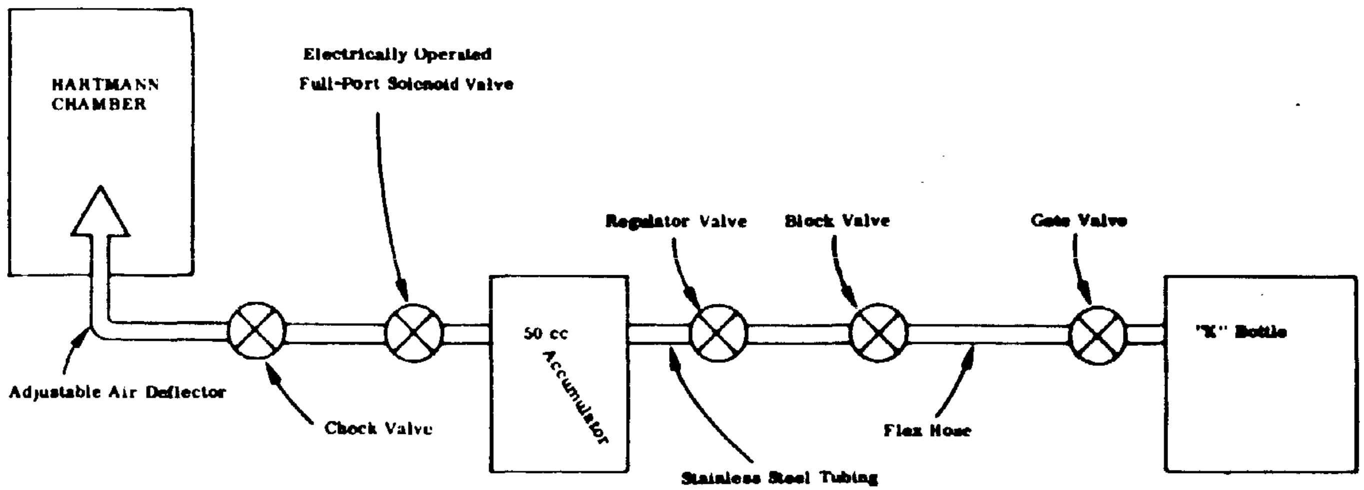


Figure 4-15. Pneumatic System for Hartmann Apparatus

- Next in line was the 50-cc air accumulator with Bourdon-type pressure gage.
- Downstream of the accumulator was the electrically operated full-port solenoid valve designed for remote operation of the valve.
- Finally, a check valve was placed between solenoid valve and chamber to prevent the combustion gases from escaping back into the dispersion reservoir.

Ignition after dust dispersal is ordinarily accomplished by connecting to the electrodes any one of the following electrical power supplies (listed in order of decreasing energy):

- Single spark discharge (50 Joules capacity) - using a Fluke 410 high voltage power supply (10,000 volt output) and a compatible capacitance bridge with a range of 10 picofarads to 1 microfarad.
- Hot wire - a 120-watt DC power supply was connected to a helical coil (1/2 inch in diameter and 3/4 inch long) made from a 15-inch length of No. 18 Nichrome V wire.
- 24-watt continuous induction spark - which consists of a capacitance discharge circuit of 1 microfarad being pulsed at 550 Hz through a high voltage transformer.

4.3.2.2 Ignition Criteria

The following visual observation criteria have been established by the Bureau of Mines in for their dust cloud ignition tests using the Hartmann Apparatus:

- Filter paper rupture - a single disc or sheet of No. 4 Whatman filter paper was held in place on top of chamber by a locking ring.
- Rupture of this disc provided evidence of ignition of the dust cloud.
- Flame propagation four inches or longer inside the tube - as observed through viewing ports inside of chamber.

4.3.2.3 Instrumentation

Two Biomation Transient Recorders were used to record the pressures of the same number of piezoelectric transducers (100 psi range) located inside the Hartmann chamber.

Due to the hazards inherent to these types of operations, the combustion chamber and the pneumatic system were isolated from the instrumentation system by reinforced concrete walls (Figure 4-16).

4.3.2.4 Procedures

4.3.2.4.1 Materials Preparation

In order to insure the accuracy of the data, the following Bureau of Mines procedures were implemented:

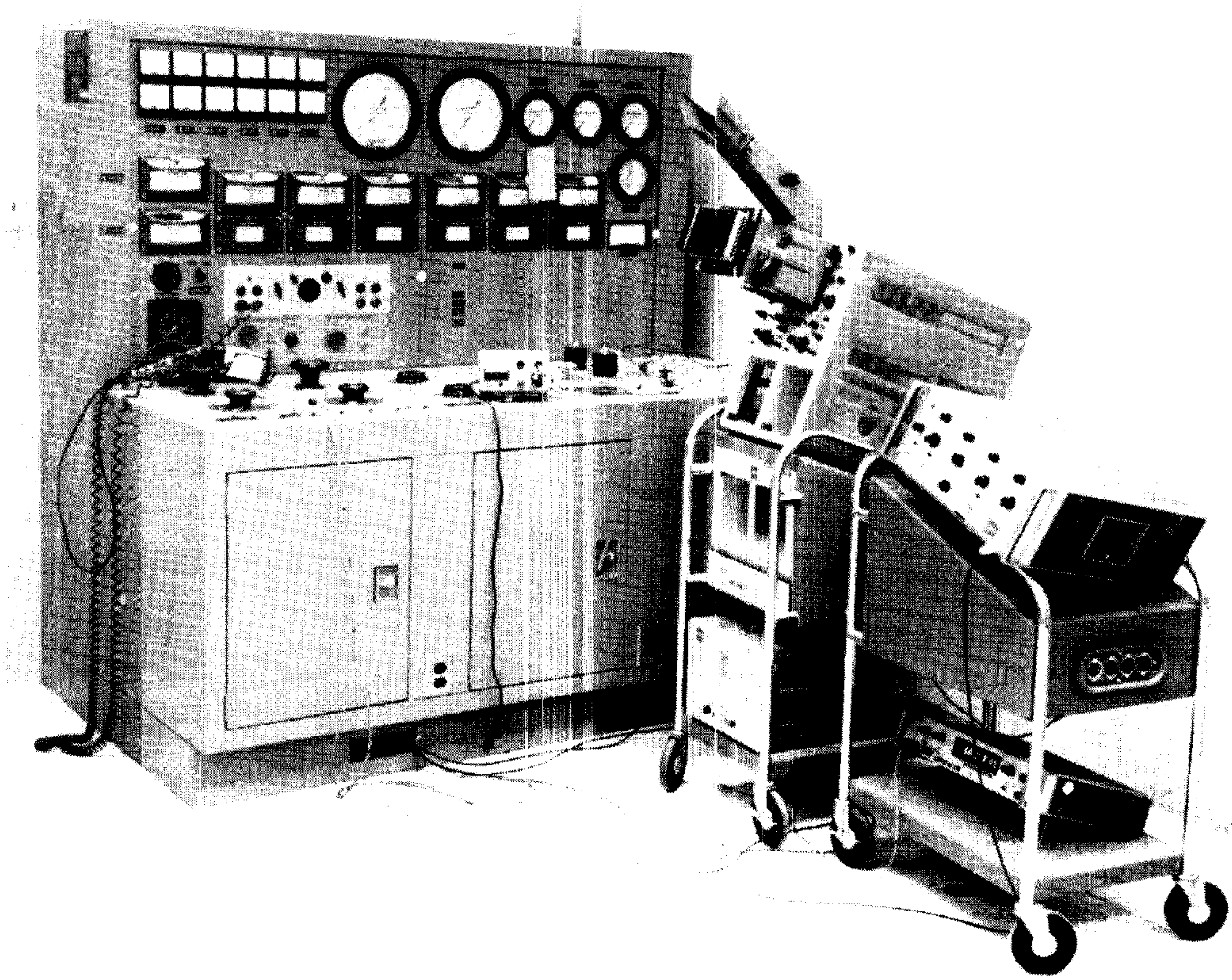


Figure 4-16. Instrumentation Control Room for Dust Testing

- To minimize particle size variance in each batch and therefore variation in test data from batch to batch, dust was sieved through No. 200 (U.S. Standard series screens).
- Sample materials were individually weighted on a triple beam balance accurate to $\pm$ 5 X 10^{-4}gm (Figure 4-17).
- All materials were dried in an oven at 75°C for 24 hours.
- As shown in Figure 4-18, the entire Hartmann Apparatus was enclosed in an air tent containing heaters to further reduce the relative humidity in and around the chamber.

4.3.2.4.2 Operation

A flow chart summarizing the Bureau of Mines procedures for evaluating the explosibility index of dusts is shown in Figure 4-19 (Phase II Final Report, GE-MTSD R-058, Appendix C).

4.3.2.4.2.1 Determination of the Minimum Density Required for Ignition. The lowest weight at which flame propagates or the minimum density required for ignition was determined as follows:

- A weighted sample was placed in the dust dispersion cup (Figure 4-14). Initially, the amount of weighted sample was determined to be at a level where a 50 percent response is expected.
- Following this, the air deflector is opened 2-1/2 turns in order to obtain optimal powder dispersal. (This adjustment was established by repeated tests and verified by the Bureau of Mines personnel.)
- With either the induction spark or hot wire ignition source operative (maintaining maximum rated voltage and current), the sample powder is then dispersed by a blast of air being admitted by the solenoid valve.
- The concentration level was moved up one step after each non-response, and down one step after each response.
- The next series of tests consisted in either moving the concentration up or down 50 percent of the previous level. This was continued until at the 5 mg concentration level, an increase in the quantity of materials failed to propagate a flame in any of four successive trials.

Results of testing using the hot wire ignition source in conjunction with 80-psi continuous air flow are given in Table 4-4, Figures 4-14 and 4-20. Also shown in Column #3 (Table 4-4) are the minimum concentration of fuels determined by the Bureau of Mines using single spark discharge ignition source.

4.3.2.4.2.2 Determination of Minimum Electrical Energy Required for Ignition. The procedure for determining the minimum electrical energy required for ignition is as follows:

Figure 4-17. Sieves and Triple Beam Balance Used for Material Preparation

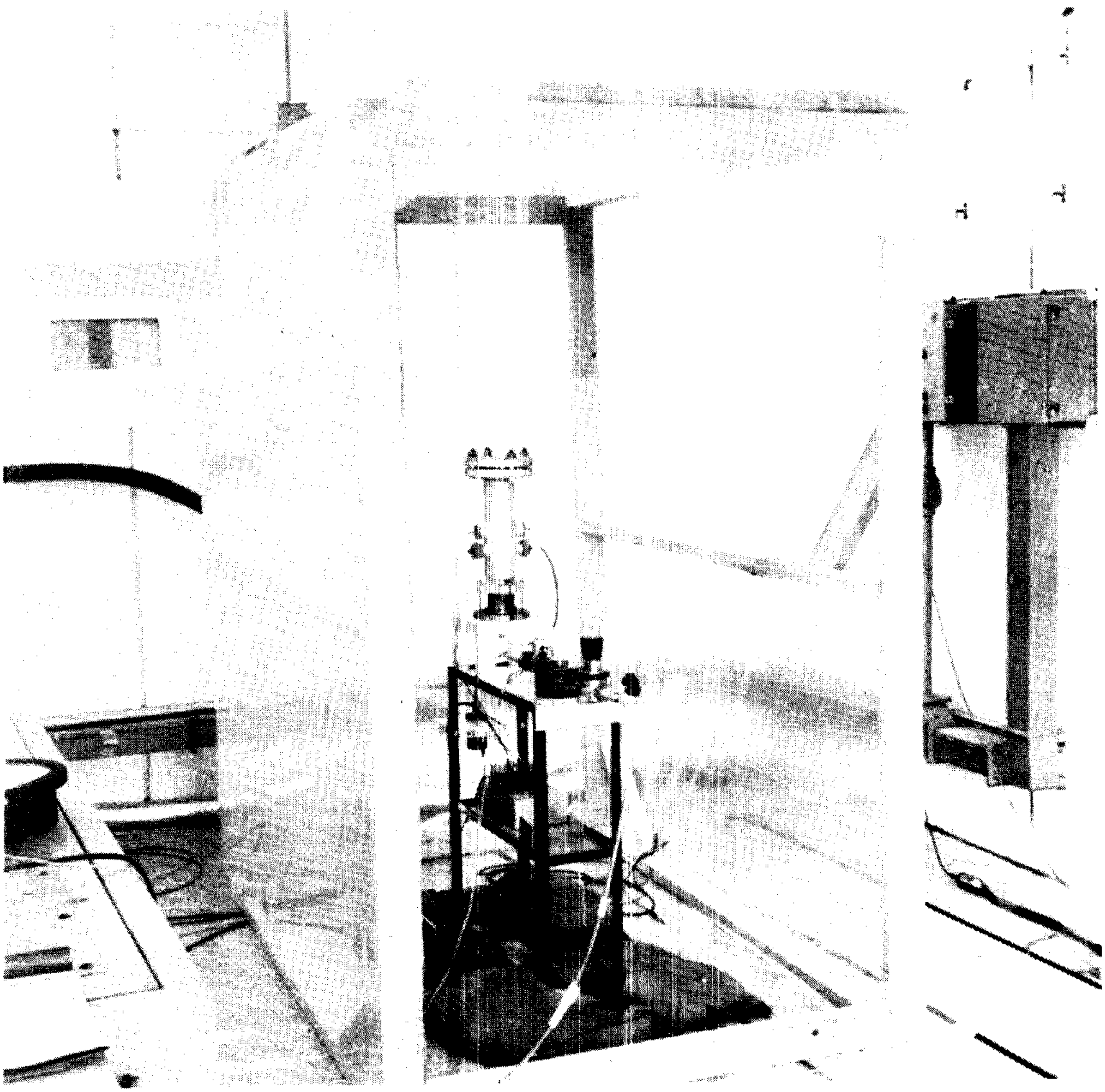


Figure 4-18. Heated Air Tent (In Order to Control the Relative Humidity)

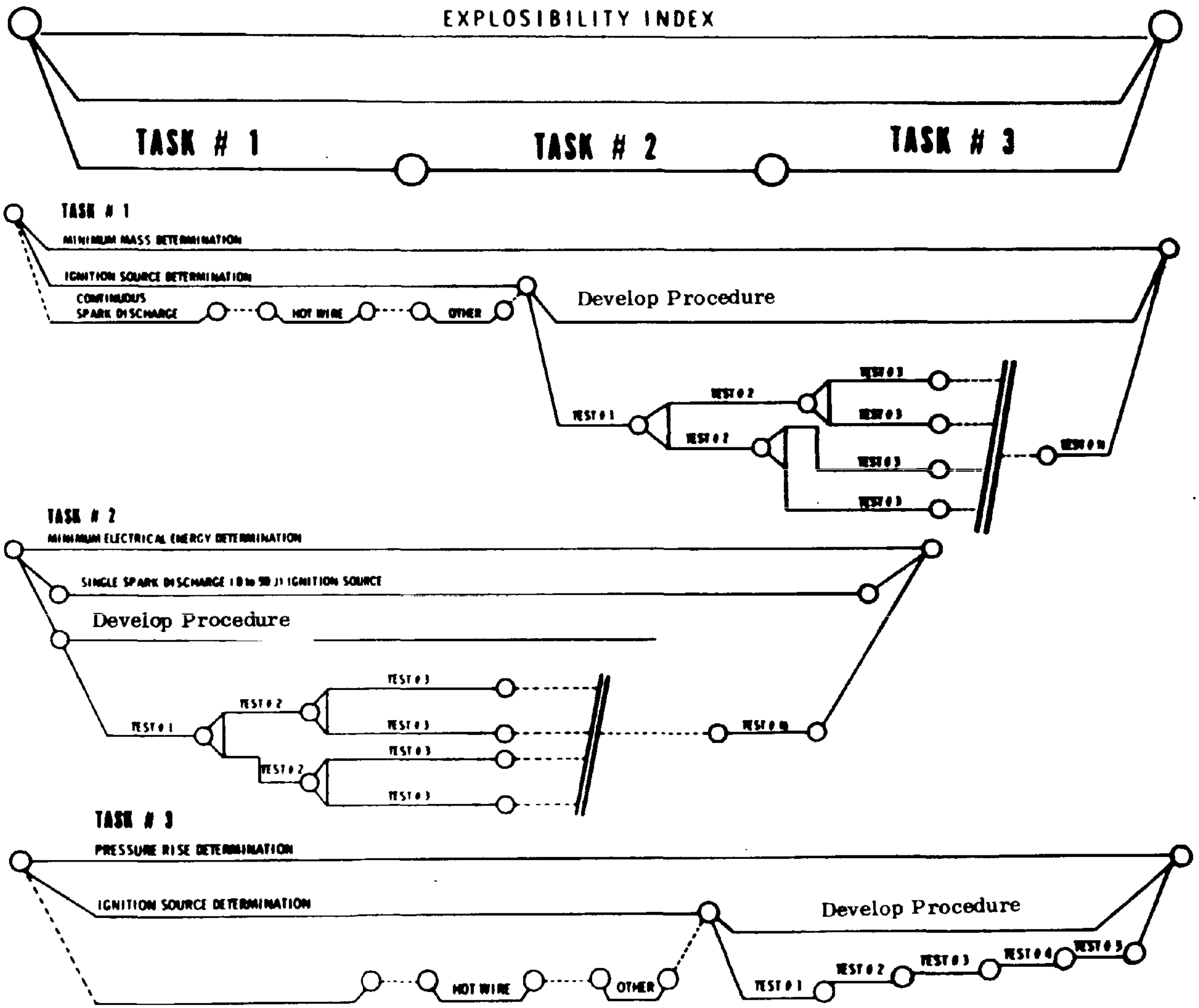


Figure 4-19. Flow Chart for Determination of Explosibility Index

Table 4-4. Summary of Results on Minimum Dust Concentration Tests (Using Hot Wire Igniter with 80 PSI Continuous Air Flow)

Material	1 Minimum Mass Required for Ignition (mg)	 ($x\ 10^{-5}$ oz)	2 Minimum Concentration (oz/ft^3)	3 Bureau of Mines* Minimum Concentration (oz/ft^3)
I Pyrotechnic Formulations				
Fuel Mix	2.0	7.05	.002	
Lactose Green	9.0	31.7	.007	
Lactose Yellow	9.0	31.7	.007	
Sulfur Yellow	10.0	35.2	.008	
Sulfur Green	31.0	109	.025	
II Fuels				
Aluminum	15	(52.9)	.013	.020
Sugar	32	(112)	.027	.045
Sulfur	131	(461)	.107	.035
Coal - Illinois	500	(1750)	.407	
Lactose	No Ignition			
Pittsburg Coal				.035

*Note: Determined using a single spark discharge ignition source.

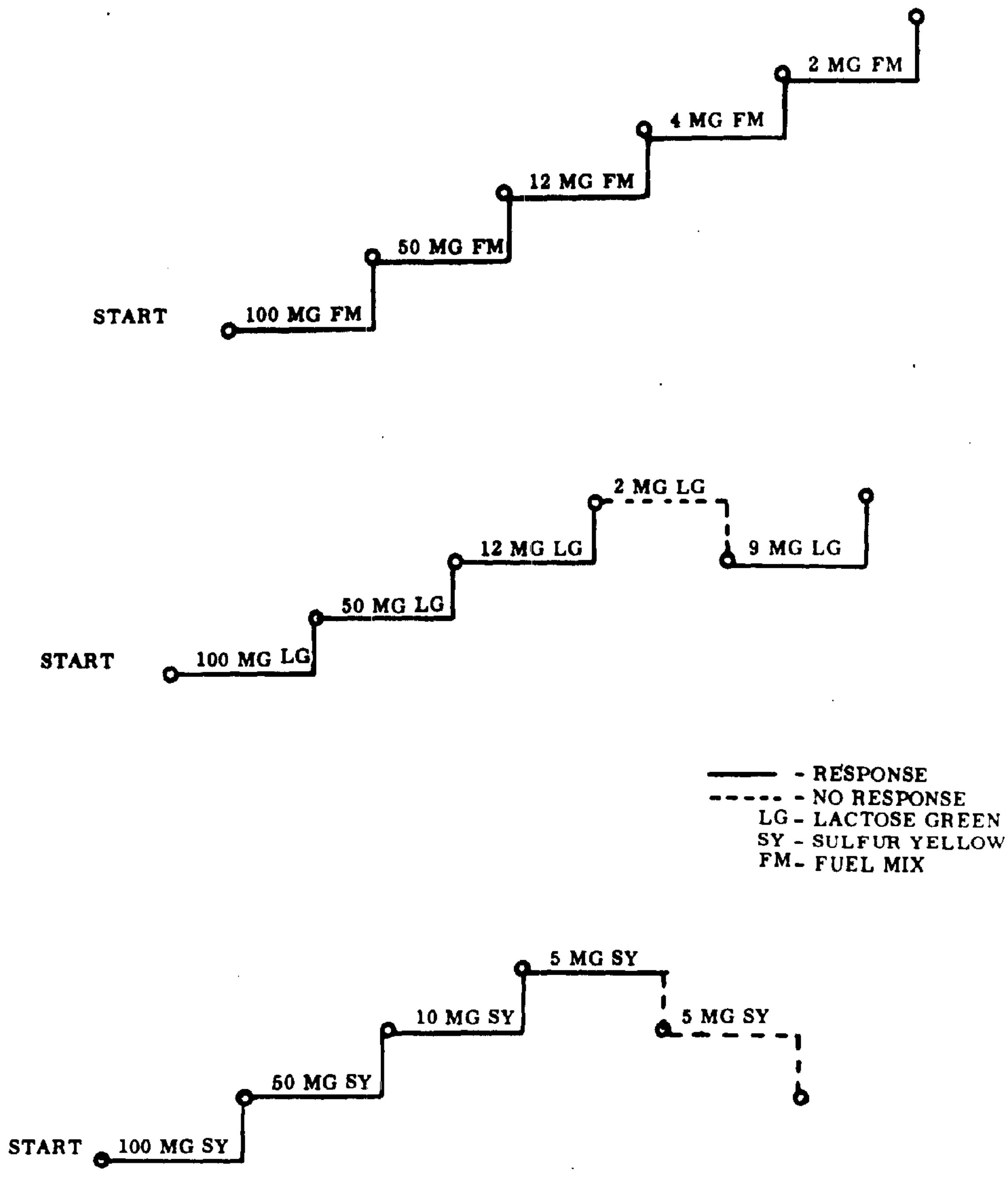


Figure 4-20. Flow Diagram for Minimum Ignition Concentration Tests (Sheet 1 of 3)

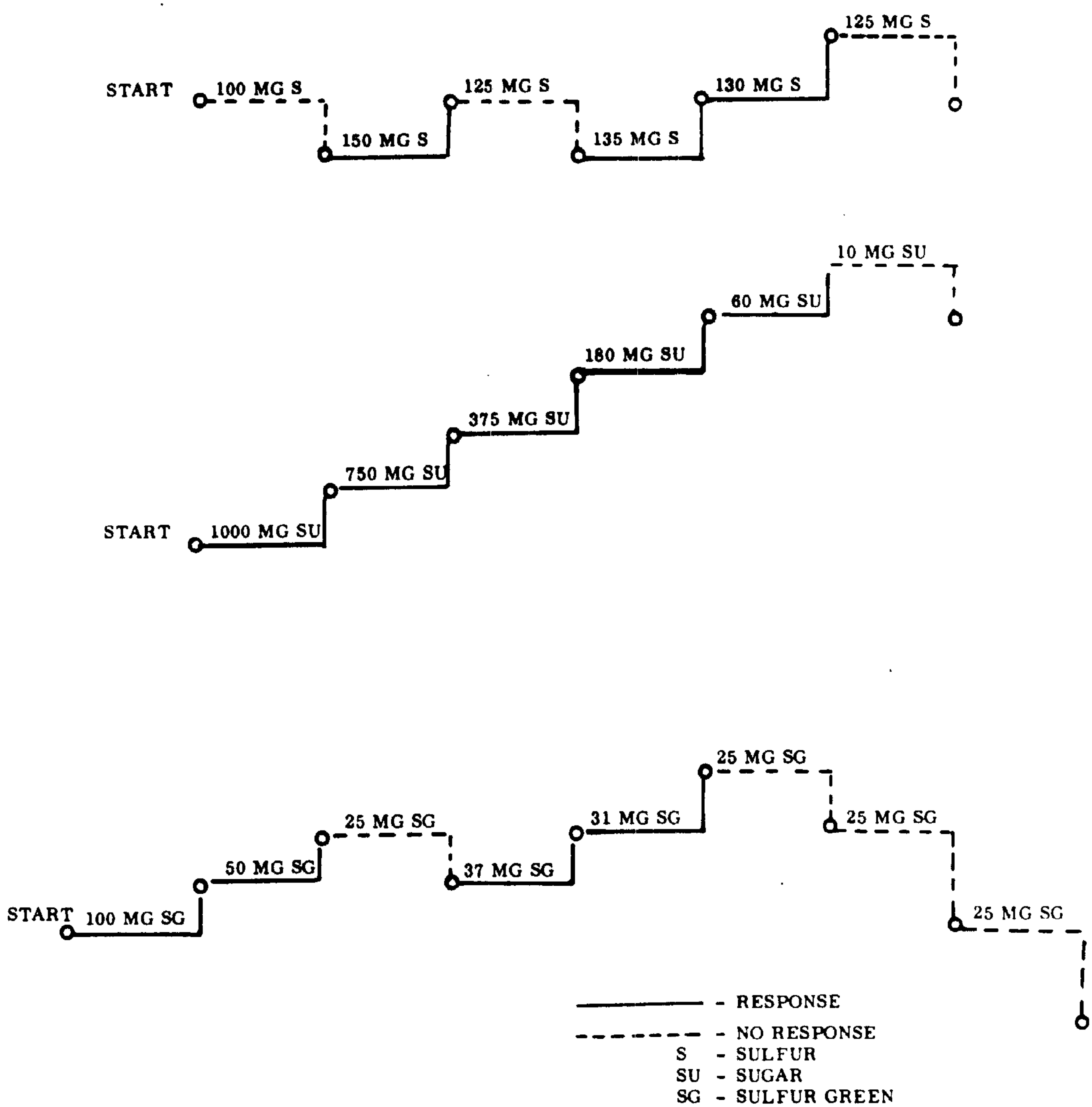


Figure 4-20. Flow Diagram for Minimum Ignition Concentration Tests (Sheet 2 of 3)

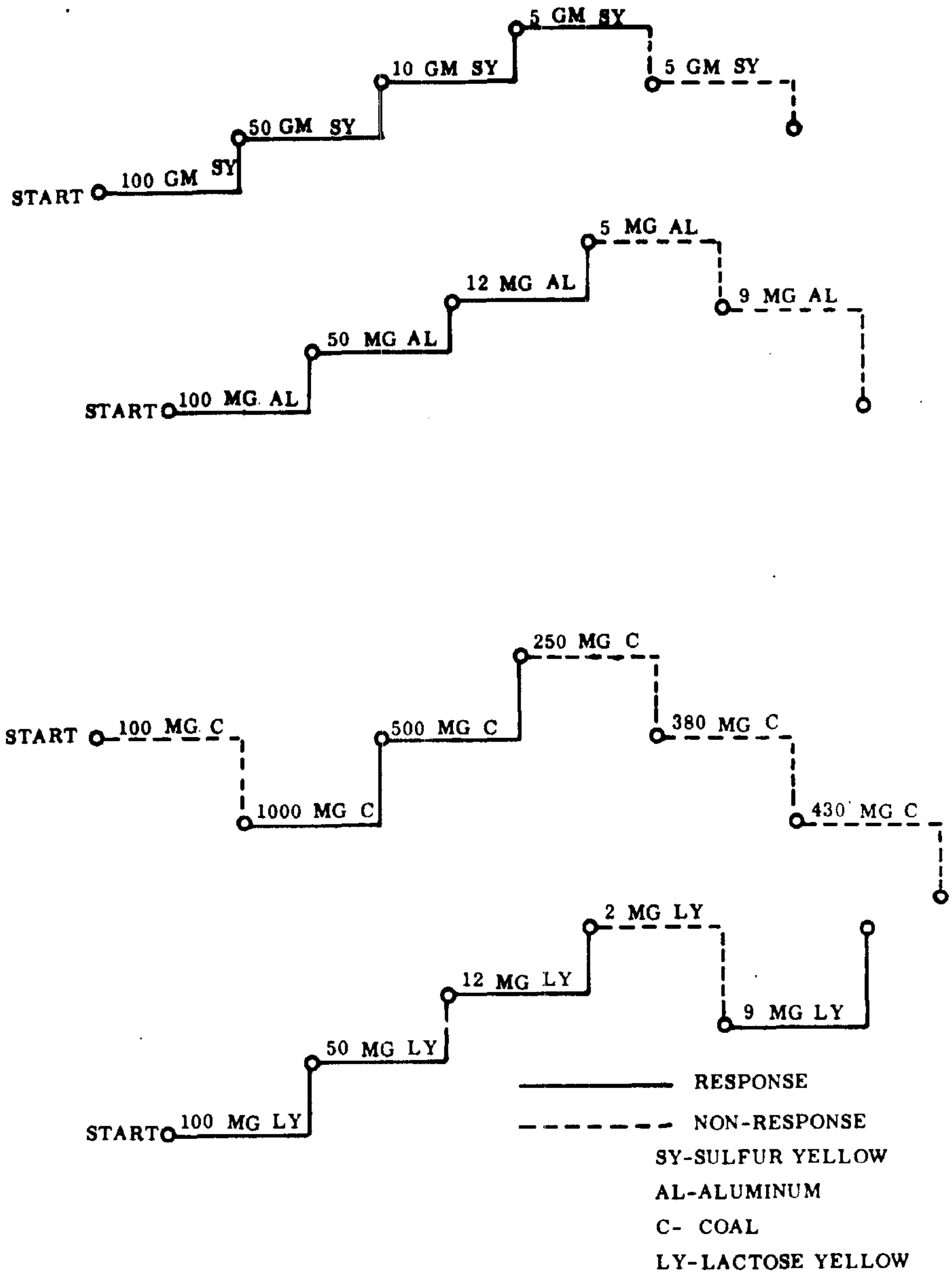


Figure 4-20. Flow Diagram for Minimum Ignition Concentration Tests (Sheet 3 of 3)

- A weighed sample was placed in the dust dispersion cup. The quantity of dust dispersed ranges from 5 to 10 times the minimum explosion concentration.
- After the weighted sample is placed in the dust dispersion cup (Figure 4-14), the deflector is opened 2-1/2 turns.
- Immediately following sample dispersal (20 milliseconds) a single spark is discharged between the electrodes (spaced approximately 1/4 inches apart).
- The energy of the spark is moved up one level after each non-response, and down one step after each response.
- The next series of tests consisted in either moving the energy level up or down 50 percent of the previous level. This was continued until at the five (5) millijoule level, an increase in the energy level failed to propagate a flame in any of four successive trials.

Normally explosion tests are made with a single burst of air at 100 psi released by the electrically operated solenoid.

4.3.2.4.2.3 Explosion Pressure and Rates of Pressure Rise. The filter paper rupture disc in the above test series is replaced with a steel cover plate in order to determine pressure and rate of pressure rise in the closed Hartmann chamber with the internally mounted piezoelectric transducer (paragraph 4.3.2.3).

- Normally, explosion tests are made at dust concentrations of 0.10, 0.20, 0.50, 1.00, and 2.00 ounce per cubic foot.
- Ignition of the dust cloud is normally produced by the continuous spark source. For dusts which ignite with difficulty, the hot coil or guncotton source is tried.

4.3.3 TEST RESULTS

4.3.3.1 Materials

4.3.3.1.1 Pyrotechnics

In order to obtain the maximum amount of information with the minimum number of tests, representative materials were tested:

- C/S Fuel Mix
- Sulfur Yellow
- Lactose Yellow
- Sulfur Green
- Lactose Green

In previous TB 700-2, TNT equivalency, DTA and Parr bomb tests, these materials proved to exhibit sensitivity and energy release values representative of the lactose, sulfur base and fuel mix smoke compositions.

4.3.3.1.2 Fuels

The following five basic fuels used in pyrotechnic munitions were tested individually:

- Coal
- Sugar
- Sulfur
- Aluminum
- Lactose

4.3.4 CONCLUSIONS AND RECOMMENDATIONS

The pyrotechnic formulations and fuels are ranked (Table 4-4) according to the minimum mass required for ignition. Under pyrotechnic formulations Fuel Mix containing no additives (dyes or inhibitors) is rated the highest followed by Lactose Green, Lactose Yellow, Sulfur Yellow, and Sulfur Green. Under fuels, aluminum is the hottest followed by sugar, sulfur, and coal. Lactose did not ignite; therefore, on the basis of this test, it would represent only a fire hazard as compared to a dust explosion hazard.

Greater reproducibility in test results was obtained using a continuous air flow in conjunction with the hot wire igniter as compared to the recommended Bureau of Mines technique consisting of a single blast of air. Deviation from recommended testing procedures was justified since it was observed that the continuous air flow operation generates greater turbulence (therefore, greater dust dispersion than the single blast technique).

- Comparison of both the single and continuous spark ignition sources with the hot wire source showed that the physical dimensions of the ignition sources greatly affect the ignition threshold or minimum amount of material required for ignition. Since it was observed that dust cloud dispersion was non-uniform, it can be concluded that the probability of ignition increases greatly with size of the ignition source. Therefore, the success of the hot wire ignition source over the spark techniques is explained in view of the large physical dimensions of the hot wire source as compared to the other spark modes. It is concluded that radiating heated surfaces (i.e., broken light bulb) can represent a more hazardous ignition source in a dust environment than spark discharge (i.e., motor brushing or frayed grounding strap).
- The Hartmann Apparatus represents a significant testing method for evaluating the sensitivity and ignition criteria of pyrotechnic dust formulations.

- The Hartmann Apparatus is useful for conducting small scale tests because the dust chamber represents at reduced scale an operational situation with all data directly relatable to a full scale accident.
- For over 15 years, the Hartmann Apparatus has been the standard method used to determine dust ignition cirteria. Data obtained under the present testing program can be directly compared to Bureau of Mines data (with appropriate modification) for the same material.
- It was observed that there was a delay of 3 - 5 seconds associated with the ignition of pyrotechnic mixes in contrast to the fuels, which ignited immediately. In view of the fact that typical pyrotechnic formulations contain 20 to 30 percent combustible fuel, a longer time is required before criteria for ignition of dusts are satisfied.
- Comparison of the minimum concentrations as obtained by the Bureau of Mines with those obtained herein show good agreement in view of the fact that different types of igniter sources were used (hot wire for data obtained herein and single spark discharge for Bureau of Mines investigations).
- Need exists for future work using the Hartmann Apparatus to determine maximum explosive pressure developed by semi-vented and completely closed chambers so as to obtain the explosive severity and run-up potential of dust reactions.
- A need exists to determine ignition criteria for dust/vapor atmosphere.
- Future work is planned that provides (cost effective) validation/replication of information required for operational shielding, suppressive construction for run-up and operational shielding applications. This will be obtained by modification of Hartmann chamber by addition of a second chamber into which suppressive/quenching materials can be inserted. Standardization of Hartmann test procedures will appear in Phase III.

4.4 HIGH EXPLOSIVE (HE) EQUIVALENCY TESTS

4.4.1 INTRODUCTION

Prior to beginning the Phase III high explosives equivalency testing program, similar tests were conducted in the Phase I program under the title of TNT Equivalency Testing. The basic purposes in both test programs were to:

a. Determine the relative energy release characteristics of pyrotechnic compositions in terms of blast overpressure, impulse, and fragmentation compared to similar characteristics exhibited by a known high explosive such as TNT or C-4.

b. Evaluate these characteristics under varying degrees of confinement, in various configurations, and with different initiating devices.

c. Determine whether, under any combination of the parameters developed in (a) and (b) above, pyrotechnics exhibit the characteristics of a detonation.

The following paragraphs chronicle the results of testing to determine these three factors.

4.4.2 HIGH EXPLOSIVES EQUIVALENCY TEST SETUP

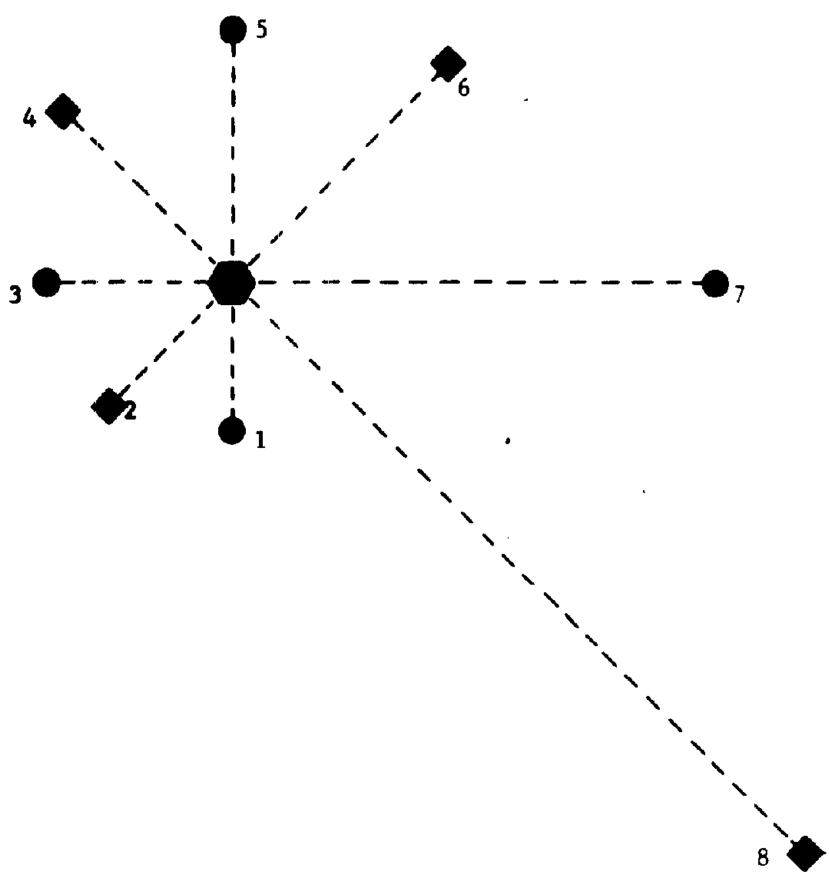
The test configuration was selected on the basis of the tests performed in the Phase I hazards evaluation program. The standard card gap pipe (1.875 inches OD x 5 1/2 inches long) specified in US Army TB 700-2 was utilized. It had the advantages of being readily available from the previous test program, offered a comparison potential to the card gap tests and was in the same L/D ratio range as the pyrotechnic end items of interest; i.e., the M-18 grenade and 105-MM canisters.

Pressure transducers were placed at distances of interest and at ranges which precluded reflected pressure waves. The distance selected varied from 2.515 feet to 13.942 feet (see Figure 4-21). The weight of 100 grams selected allowed the test vessel to be loaded to a standard geometric configuration approximating a 2.5:1 cylindrical shape. Effects of sample shape and charge weight are discussed later in this report.

The hardware and materials utilized in this test series were as follows:

- Test fixture as shown in Figure 4-22, 4-23, and 4-24
- J-2 engineers special electric blasting cap
- Sample material
- Blast measurement system

TXDCR No.	DIST. Ft., In.
1	2'5-½"
2	2'11"
3	3'4-1/16"
4	4'3/4"
5	4'6-11/16"
6	5'4-1/16"
7	8'2-15/16"
8	13'10-3/8"



SYMBOLS:

TEST CHARGE

TXDCR/OSCILLOSCOPE SYSTEM

TXDCR/RECORDER SYSTEM

Figure 4-21. Transducer Array for HE Equivalency Tests

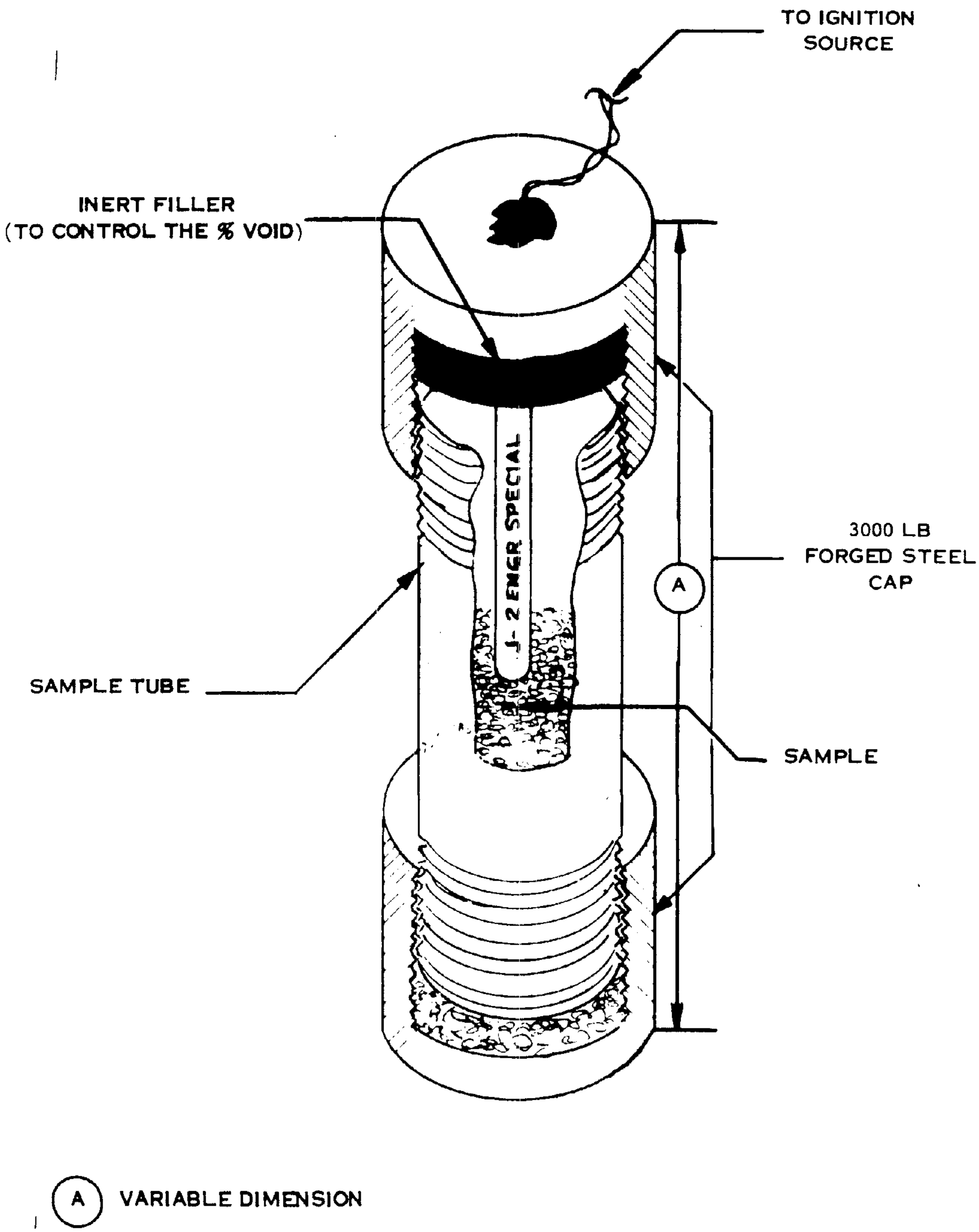


Figure 4-22. HE Equivalency Test Vessel Showing Cut-A-Way View

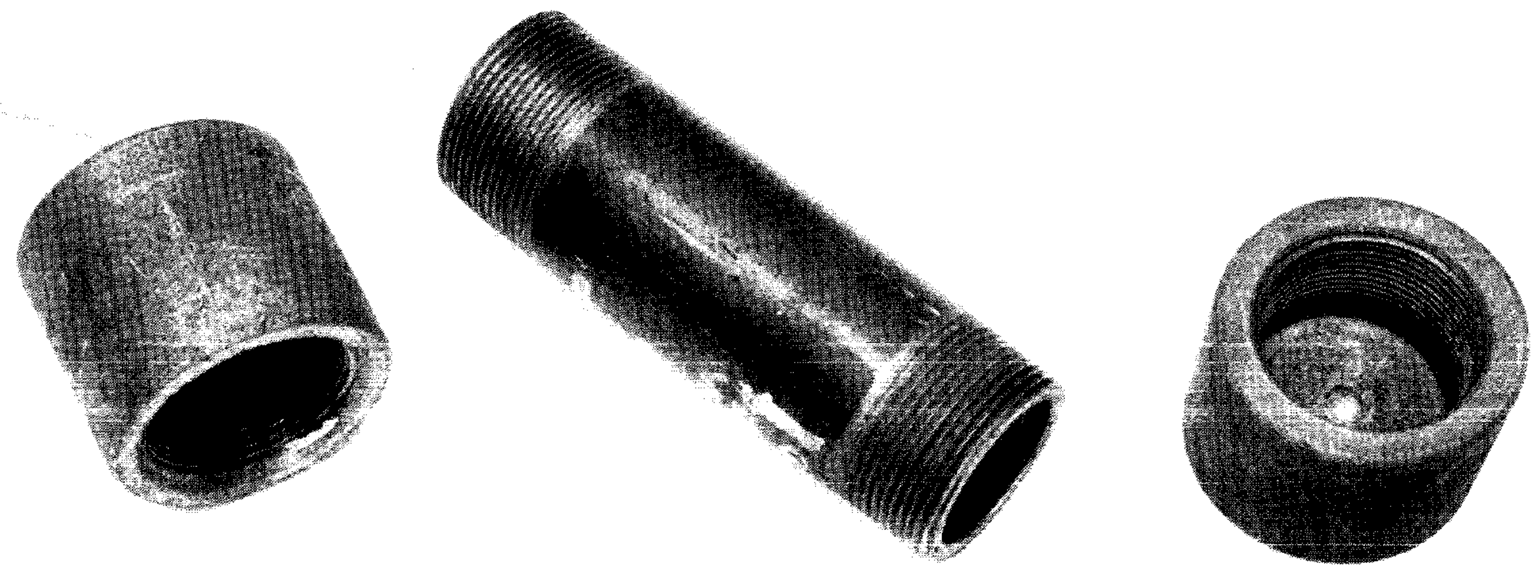


Figure 4-23. HE Equivalency Test Vessel Disassembled

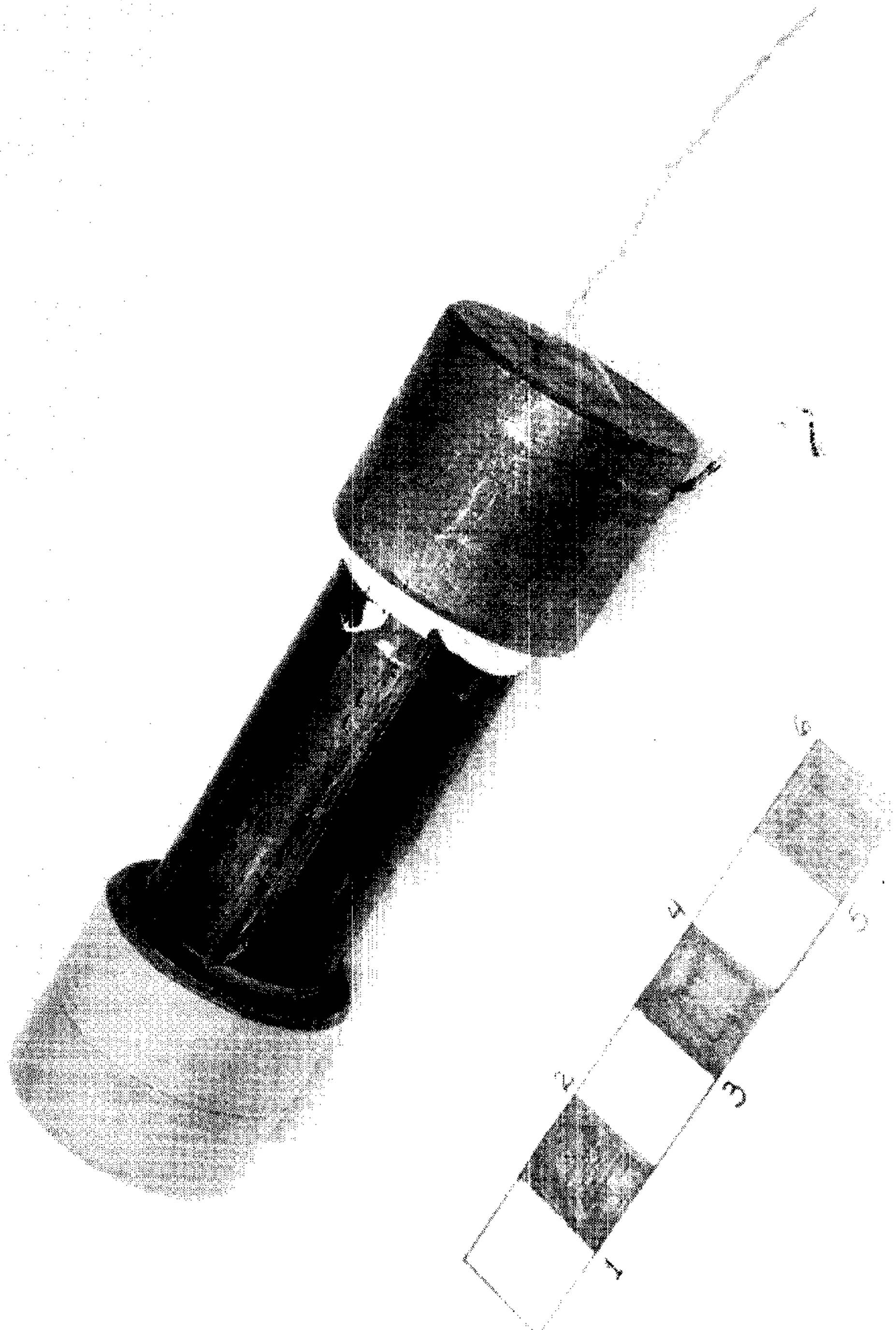


Figure 4-24. HE Equivalency Test Vessel Assembled

The blast instrumentation system used in this program is shown in Figures 4-25 and 4-26 and consisted of the following:

- Piezoelectric Transducer - which emits a signal that is a function of the magnitude of the overpressure. Since it is a dynamic instrument, it requires no external overpressure excitation potential.
- Source Follower - an integrated circuit that is directly coupled to the piezoelectric transducer and converts the charge signal from the transducer to voltage signals suitable for memory of the biomation transient recorders.
- Charge Amplifier - a solid-state unit which converts charge signals from the piezoelectric transducer to voltage signals suitable for display on oscilloscopes.
- Peak Meter - which indicates the voltage signal encountered from the blast overpressure signal.
- Transient Recorders - which utilize a very high speed six-bit analog to digital converter with a maximum word conversion rate of 10 mHz combined with a 6 bit x 128 word MOS shift register memory to capture and hold the digital equivalent of the analog signal from the transducer. This signal is then displayed on an X-Y plotter to be converted into engineering units for data reduction of a blast overpressure and impulse readings.
- Oscilloscope - which is set for a single sweep external trigger and is triggered by the machine on the positive rise of the firing pulse. The oscilloscope records blast overpressure utilizing the Polaroid camera pack.
- Electronic Counter - which is triggered by a break wire to record time of arrival of the shock front of the blast overpressure at each transducer.
- X-Y Plotter - which is an analog device that graphically displays the blast overpressure held in memory by the transient recorder. The graphic display is then converted into engineering units for further data reduction.

The equipment utilized for the blast overpressure instrumentation system consisted of the following:

- Eight, Susquehanna Instrument Company Model ST-7, Piezoelectric Transducers.
- Four, PCB Piezotronics Inc., Model 401A11 ICP, Source Followers.
- Four, Kistler Model 504A, Charge Amplifiers.
- Two, Kistler Model 538A, Peak Meter Indicators.
- Four Type 502A, Dual-beam Oscilloscopes with Camera Packs.

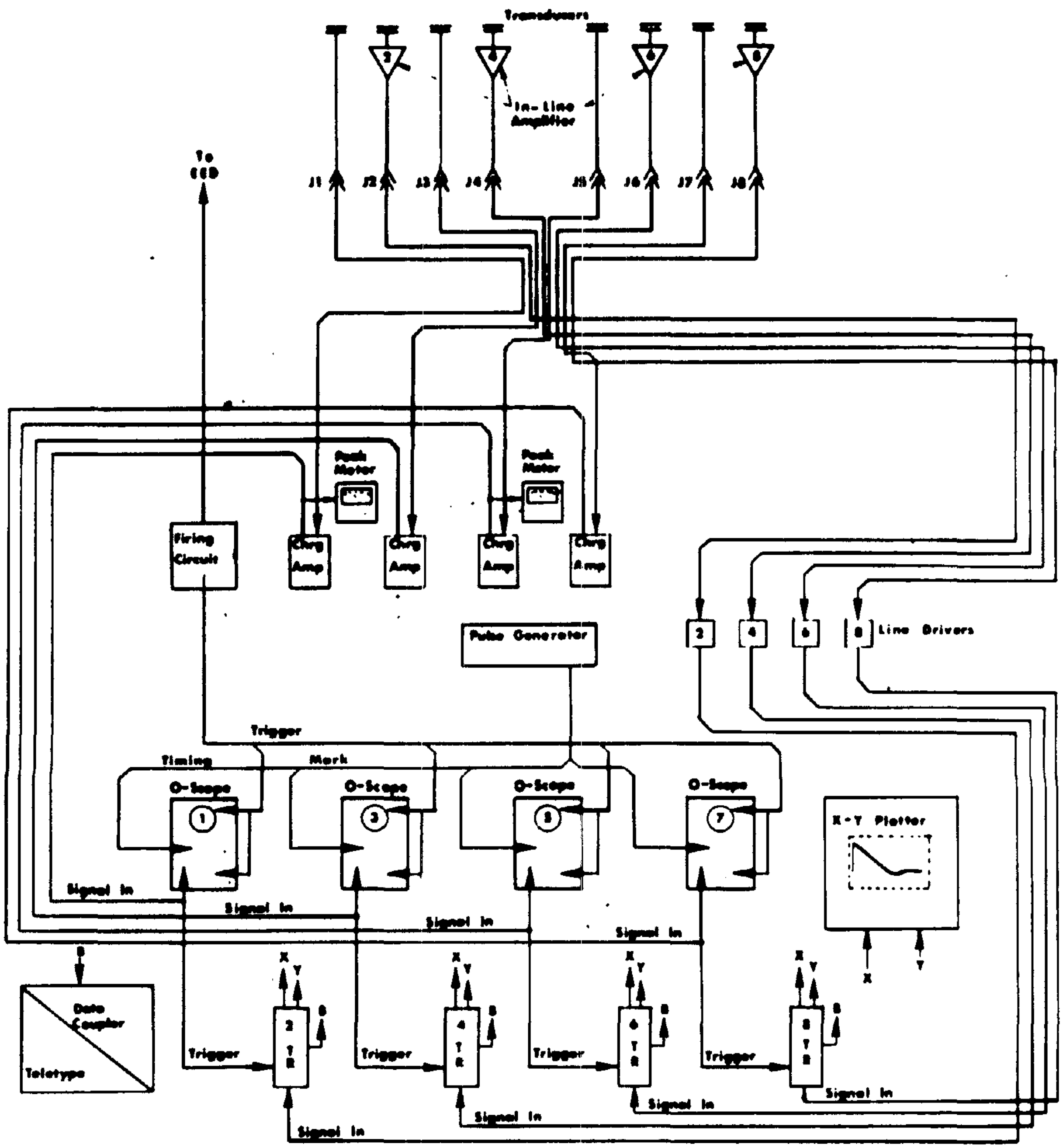


Figure 4–25. Blast Instrumentation System

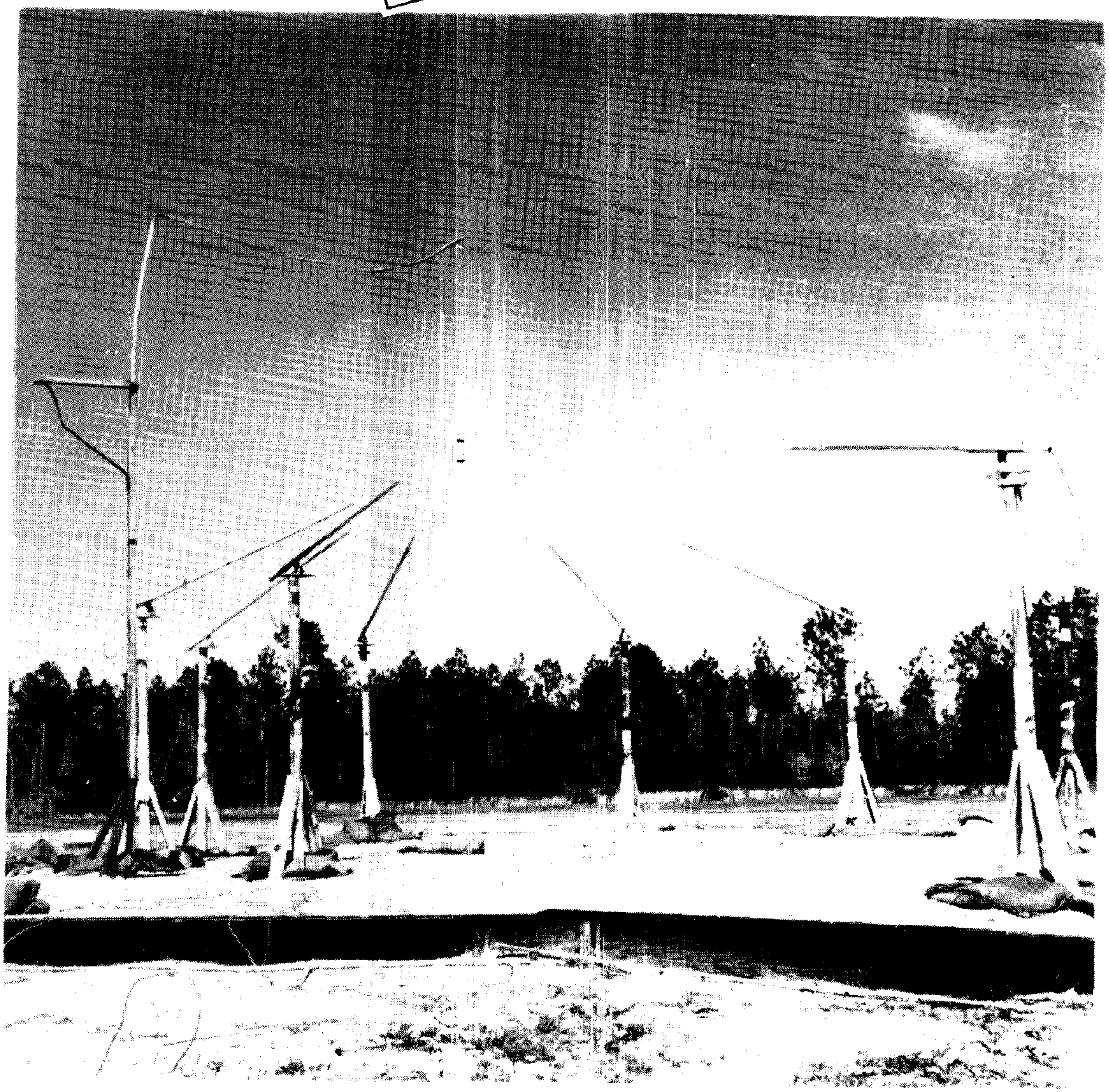


Figure 4-26. Typical Transducer Array - HE Equivalency Test Setup

- One, Hewlett-Packard Model 2501C, Digital Voltmeter.
- One, Hewlett-Packard 5233L, Electronic Counter.
- One, DuPont Model CD-12, Blasting Machine or equivalent.
- One Firing Circuit Voltage Divider (as-built).
- One X-Y Plotter.

The deployment of the sample container or vessel was as follows:

a. Place the material in the vessel.

b. Place the cap (with a preinstalled J-2 blasting cap) on the threaded pipe and tighten securely.

c. Suspend the loaded fixture in the center of the instrumented test area.

d. Initiate the test observing appropriate safety precautions.

4.4.3 TNT EQUIVALENCY DATA RATIONALE

The evaluation of the values received for peak pressure, function time, positive duration and impulse from the detonation of TNT and C-4 in both free air and confined modes provides a basis for comparison for pyrotechnics. Evaluation of blast measurement data requires the establishment of a common denominator against which the data is compared. The standard normally used is the free air spherical pentolite data contained in Goodman's BRL Report 1092, February 1960 and Soroka's computerized tabulation of the same data. While most TNT (or HE) equivalency data is compared to spherical pentolite, it was determined that for the purpose of this report that additional independent data curves for two high explosive materials (TNT and C-4) would be developed, thereby allowing comparison of pyrotechnic test data to other HE equivalency data.

It was further decided to conduct HE calibration tests and subsequently construct reference curves on the following basis:

a. Free air tests, wherein explosive charges were suspended from a support of sufficient height to eliminate reflected pressure. The HE materials were packaged in cardboard tubes for minimum confinement of the material. The dimensions of the tube were chosen to maintain the same L/D ratio as the standard card gap sample, and the confinement test vessels.

b. Confined, wherein the explosive charge was suspended as before and confined in the vessel described in paragraph 4.4.2.

The material selected for development of standard or reference curves included flaked TNT and C-4 in weights of 50, 75, 100 and 125 grams.

4.4.4 REFERENCE AND CALIBRATION DATA

4.4.4.1 Overpressure

The high explosives reference and calibration data was derived by firing no less than five charges at each of the weights (50, 75, 100, and 125 grams) of each of the candidate materials (TNT and C-4) in free air and in the confined state. The data in digital form as processed by the CSC 930 computer is shown in Figures 4-27 through 4-30. The data is displayed as follows (starting with the right hand column):

- The first column identifies the test
- The next eight columns show peak pressure and impulse for each transducer channel in terms of the scaled distance Z.
- Fof each test series (an HE weight), a mean value and standard deviation for peak pressure and impulse are calculated.

The digital data was plotted by a Stromberg-Carlson 4020 Plotter and is shown in Figures 4-31 through 4-38.

For each set of HE data, a computer plot of the data points is shown followed by the same plot with the curves of primary interest superimposed over them. The curves of primary interest are:

- Curve 1 - Flake TNT charges of 50, 75, 100 and 125 grams detonated in the confined state.
- Curve 2 - Flake TNT charges of 100 grams detonated in the confined state.
- Curve 3 - C-4 charges of 50, 75, 100 and 125 grams detonated in the confined state.
- Curve 4 - C-4 charges of 100 grams detonated in the confined state.

Of prime significance in these plots is the fact that flake TNT confined exhibits higher peak pressure values than in free air. This is due to the fact that a portion of the flake TNT, being in a granular state, does not enter into the detonation reaction, but merely scatters due to the reaction of the flashing cap and the portion of the TNT that does react. Conversely, C-4 detonated in the confined state exhibits lower peak pressures than when detonated in free air. This is due to the fact that some of the energy release by the confined C-4 is used in the rupturing of the confining vessel. From this one can postulate that for high explosives calibration and standarization work, condensed explosives confined in the same medium as the material of interest should be used.

TRINITROTOLUENE (TNT) FREE AIR DETONATION

WEIGHT 50.00 GRAMS

CHANNEL	1		2		3		4		5		6		7		8	
Z VALUES	5.25		6.25		7.13		8.63		9.67		11.29		17.34		29.85	
	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP
4-1-02	9.10	.00	6.00	1.40	6.30	.00	3.00	.93			2.10	.71	1.38	.00	.62	.27
4-1-03	9.90	.00	5.30	1.43	5.04	.00	2.90	.88	2.92	.00	1.50	.67	1.38	.00	.52	.21
4-1-04	11.06	.00	4.80	1.37	4.74	.00	2.80	.85	2.72	.00	1.60	.60	1.44	.00		
4-1-05	10.45	.00	5.30	1.38	4.06	.00	2.80	.91	2.80	.00	2.55	.79	1.44	.00	.57	.24
4-1-06	9.66	.00	4.70	1.25	4.50	.00	2.00	.84	2.60	.00	2.40	.77	1.41	.00	.60	.25
4-1-13	8.40	.00	4.00	1.00	4.50	.00	2.60	.71	2.70	.00	1.90	.93	1.32	.00	.40	.17
MEAN	9.91	.00	5.02	1.30	4.99	.00	2.82	.85	2.75	.00	1.94	.69	1.39	.00	.54	.23
STAN DEV	1.02	.00	.60	.16	.60	.00	.13	.08	.12	.00	.47	.09	.05	.00	.09	.04

TRINITROTOLUENE (TNT) FREE AIR DETONATION

WEIGHT 75.00 GRAMS

CHANNEL	1		2		3		4		5		6		7		8	
Z VALUES	4.59		5.46		6.23		7.54		8.45		9.87		15.15		25.38	
	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP
10-1-01			10.20	2.41			5.10	1.60	3.20	.00	2.70	1.15	3.04	.00		
10-1-02			7.20	2.06							2.80	1.14				
10-1-03	10.10	.00	7.80	1.89	7.60	.00			3.50	.00	2.40	1.41	2.80	.00		
10-1-04	9.00	.00	7.00	1.88					4.00	.00	2.80	1.13	2.88	.00	.76	.00
10-1-05	11.80	.00	7.80	2.20	7.20	.00	4.35	1.31	5.10	.00	3.52	1.42	4.56	.00		
10-1-06	9.00	.00	7.50	1.94	6.00	.00	3.90	.81			3.36	1.26	3.28	.00		
10-1-07	11.70	.00	7.06	1.98	7.60	.00	4.35	1.12			2.61	1.11	3.00	.00	.56	.33
10-1-08	11.88	.00			7.40	.00	3.90	1.54	4.90	.00	2.96	1.23	3.28	.00	.54	.33
10-1-09	10.35	.00	7.80	2.19			4.95	1.35	4.90	.00	3.20	1.35	3.36	.00	.53	.29
10-1-10	9.90	.00	6.40	1.93	7.20	.00	4.65	1.60	5.00	.00	3.04	1.26	3.20	.00	.50	.32
10-1-11	10.00	.00	7.60	1.98	8.00	.00	4.00	1.67	5.00	.00			3.00	.00	.55	.32
MEAN	10.51	.00	7.81	2.05	7.29	.00	4.50	1.37	4.45	.00	2.96	1.25	3.24	.00	.59	.26
STAN DEV	1.14	.00	.94	.18	.63	.00	.45	.29	.77	.00	.35	.12	.50	.00	.09	.13

TRINITROTOLUENE (TNT) FREE ATR DETONATION

WEIGHT 100.00 GRAMS

CHANNEL	1		2		3		4		5		6		7		8	
Z VALUES	4.17		4.96		5.66		6.85		7.68		8.97		13.76		23.06	
	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP
50-0-25	13.20	.00	9.52	2.42	7.60	.00			4.80	.00					2.02	.85
50-0-26	13.00	.00	9.24	2.72	8.00	.00	6.20	1.81	6.00	.00	3.42	1.29	2.20	.00	1.95	.87
50-0-27	13.50	.00	10.64	2.75	9.04	.00	6.20	1.79	5.50	.00	3.70	1.36	2.50	.00	1.92	.84
50-0-28	12.75	.00	8.75	2.45	8.00	.00	4.80	1.58	5.00	.00	3.80	1.27	2.40	.00	1.98	.84
50-0-29	11.00	.00					4.40	1.58	5.00	.00	2.80	1.04	2.20	.00	1.90	.78
MEAN	12.85	.00	9.54	2.58	8.16	.00	5.40	1.69	5.26	.00	3.43	1.24	2.34	.00	1.93	.84
STAN DEV	1.10	.00	.80	.17	.62	.00	.94	.13	.49	.00	.45	.14	.13	.00	.04	.03

TRINITROTOLUENE (TNT) FREE AIR DETONATION

WEIGHT 125.00 GRAMS

CHANNEL	1		2		3		4		5		6		7		8	
Z VALUES	3.87		4.61		5.25		6.36		7.13		8.32		12.78		21.41	
	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP
0-0-10	24.70	.00	10.20	2.53			6.64	2.17	6.00	.00			2.25	.00	2.15	.94
0-0-11	23.40	.00	9.90	2.42	9.20	.00	6.40	2.07	6.00	.00			2.50	.00	2.50	1.01
0-0-12	28.60	.00	11.70	2.94	10.80	.00	7.40	2.31	6.50	.00			2.36	.00	2.55	1.34
0-0-13	21.40	.00	9.90	2.41	9.60	.00	6.70	2.15	6.00	.00			2.30	.00	2.15	.90
0-0-14	23.90	.00	10.60	2.30	10.40	.00	6.00	1.87	5.90	.00			2.36	.00	2.25	1.33
MEAN	24.40	.00	10.46	2.52	10.00	.00	6.63	2.12	6.00	.00000.00000.00			2.35	.00	2.32	1.12
STAN DEV	2.64	.00	.75	.25	.73	.00	.51	.16	.24	.00	.00	.00	.09	.00	.19	.20

Figure 4-27. Computer Print-out of Overpressure and Impulse – TNT Free Air HE Equivalency Tests

TRINITROTOLUENE (TNT) CONFINED DETONATION

WEIGHT 50.00 GRAMS

CHANNEL	1		2		3		4		5		6		7		8	
Z VALUES	5.25		6.25		7.13		8.03		9.67		11.29		17.34		29.05	
	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP
34-0-01			7.90	1.30	12.50	.00	7.70	2.32	8.60	.00	3.15	1.30	1.70	.00	.42	.31
34-0-02	18.70	.00	9.40	1.00	11.30	.00	6.40	1.09	7.60	.00	2.70	1.00	1.70	.00	.54	.30
34-0-03	16.50	.00	7.20	1.37	11.00	.00	6.40	2.44	6.00	.00	3.70	1.22	2.10	.00	.57	.43
34-0-04	17.00	.00	7.60	1.53	12.50	.00	6.50	1.09			3.40	1.19	2.10	.00	.42	.31
34-0-05	16.50	.00	11.60	2.00	15.50	.00	9.80	3.28	8.40	.00	3.70	1.19	1.80	.00	.57	.46
MEAN	17.17	.00	8.74	1.60	12.56	.00	7.36	2.37	7.65	.00	3.34	1.20	1.80	.00	.50	.38
STAN DEV	1.34	.00	1.80	.29	1.70	.00	1.47	.57	1.10	.00	.42	.00	.20	.00	.08	.07

TRINITROTOLUENE (TNT) CONFINED DETONATION

WEIGHT 75.00 GRAMS

CHANNEL	1		2		3		4		5		6		7		8	
Z VALUES	4.59		5.46		6.23		7.54		8.45		9.87		15.15		25.38	
	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP
34-0-06	29.50	.00	15.60	3.10	16.00	.00	7.80	2.58	8.00	.00	4.50	1.80	2.50	.00	.72	.60
34-0-07	35.50	.00	9.00	1.82	10.50	.00	6.00	1.62	9.10	.00	5.70	2.29	3.20	.00	.72	.65
34-0-08	33.80	.00	16.00	3.03	16.80	.00	8.00	3.01	7.60	.00	4.45	1.66	2.40	.00	.52	.44
34-0-09	27.90	.00	15.00	3.07	16.50	.00	8.20	3.07	8.00	.00	4.70	1.80	2.60	.00	.45	.46
34-0-10	30.50	.00	11.60	2.54	14.30	.00	8.30	3.19	9.50	.00	4.20	.87	2.90	.00	.62	.56
MEAN	31.44	.00	13.60	2.71	14.82	.00	7.82	2.69	8.60	.00	4.71	1.70	2.72	.00	.61	.54
STAN DEV	3.13	.00	3.15	.55	2.60	.00	1.00	.64	.70	.00	.50	.52	.33	.00	.12	.09

TRINITROTOLUENE (TNT) CONFINED DETONATION

WEIGHT 100.00 GRAMS

CHANNEL	1		2		3		4		5		6		7		8	
Z VALUES	4.17		4.96		5.66		6.85		7.60		8.97		13.76		23.06	
	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP
50-0-01	57.60	.00	16.80	3.41	16.00	.00	7.90	2.72	6.60	.00	3.40	1.66				
50-0-02	43.20	.00	14.70	2.89	15.00	.00	8.00	2.70	6.20	.00	3.60	1.56	3.40	.00		
50-0-03	30.20	.00	14.70	2.32	10.20	.00	7.60	2.39	6.50	.00	4.05	1.80	3.50	.00	2.50	1.21
50-0-30	27.50	.00	11.60	2.89	14.00	.00	9.40	3.14	9.00	.00	5.00	2.14	3.40	.00	2.35	1.00
50-0-31	30.50	.00	14.70	3.61	14.40	.00	8.80	2.87	8.50	.00	5.50	2.10	3.70	.00	2.70	1.21
MEAN	37.82	.00	14.50	3.02	13.92	.00	8.50	2.76	7.36	.00	4.31	1.87	3.50	.00	2.52	1.17
STAN DEV	12.62	.00	1.86	.51	2.21	.00	.73	.27	1.29	.00	.91	.20	.14	.00	.18	.08

TRINITROTOLUENE (TNT) CONFINED DETONATION

EIGHT 125.00 GRAMS

ANNEL	1		2		3		4		5		6		7		8	
ALUES	3.87		4.61		5.25		6.36		7.13		8.32		12.70		21.41	
	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP
50- -04			21.60	5.24	16.00	.00			7.30	.00						
50- 05			17.10	4.28	15.20	.00	9.60	2.97	9.20	.00			3.20	.00		
0-0 06			16.20	4.29	14.80	.00	9.60	3.69	8.00	.00	6.60	2.25	3.90	.00		
0-0 7			16.20	4.43	16.00	.00	11.20	4.03	9.50	.00	7.30	2.57				
0-0 08			19.20	4.54	15.20	.00	9.60	.00	8.50	.00						
0-0-09	22.10	.00	12.00	3.09	8.00	.00	7.20	1.96	6.00	.00						
11-1-13			16.20	4.67	14.40	.00	9.60	3.90	8.80	.00	6.60	2.72	3.30	.00	2.05	1.10
11-1-14	34.06	.00	15.30	4.10	15.60	.00	11.20	3.90	10.00	.00	7.35	3.26	3.50	.00	1.90	1.37
11-1-15	33.80	.00	16.20	4.60	12.60	.00	10.50	4.57	8.00	.00	7.35	4.49	3.30	.00	1.95	1.66
MEAN	29.99	.00	16.67	4.37	14.38	.00	9.81	3.14	8.37	.00	7.04	3.06	3.44	.00	1.97	1.41
STAN DEV	6.83	.00	2.63	.58	2.39	.00	1.27	1.50	1.22	.00	.40	.80	.20	.00	.08	.25

Figure 4-28. Computer Print-out of Overpressure and Impulse - TNT Confined HE Equivalency Tests

COMPOSITION C-4 (C-4) FREE AIR DETONATION

WEIGHT 50.00 GRAMS

CHANNEL	1		2		3		4		5		6		7		8	
Z VALUES	5.25		6.25		7.13		8.63		9.67		11.29		17.34		29.05	
	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP
3-1-07	24.30	.00	10.20	2.94	12.00	.00	7.30	2.30	6.30	.00	4.00	1.55	2.80	.00	1.20	.78
3-1-08	30.60	.00	13.90	3.57	10.00	.00	10.35	2.91	9.00	.00	5.00	2.02	3.50	.00	1.42	.93
3-1-09	36.00	.00	15.00	4.09	15.50	.00	8.00	2.70	7.50	.00	4.64	1.90	3.60	.00	1.40	1.05
3-1-10	24.30	.00	11.16	2.90	12.50	.00	7.35	2.34	7.20	.00	4.32	1.64	3.00	.00	1.22	.81
4-1-12			16.00	4.13	15.00	.00	7.10	2.76	7.50	.00	5.60	2.37	3.40	.00	1.12	.85
MEAN	28.80	.00	13.27	3.52	14.76	.00	8.04	2.61	7.90	.00	4.72	1.91	3.20	.00	1.28	.89
STAN DEV	5.64	.00	2.49	.60	2.24	.00	1.34	.25	.97	.00	.62	.33	.35	.00	.16	.11

COMPOSITION C-4 (C-4) FREE AIR DETONATION

WEIGHT 75.00 GRAMS

CHANNEL	1		2		3		4		5		6		7		8	
Z VALUES	4.59		5.46		6.23		7.54		8.45		9.87		15.15		25.30	
	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP
3-1-01			19.20	5.05			11.40	3.70			9.44	3.96			1.92	.04
3-1-02	36.30	.00	18.40	5.05	20.50	.00	12.10	3.09	12.00	.00	8.44	3.35				
3-1-03	40.50	.00	18.90	5.01	21.50	.00	11.05	3.58	12.00	.00	7.44	2.98	4.30	.00	1.84	1.42
3-1-04	42.30	.00	17.20	4.50	22.50	.00	11.40	3.67	10.20	.00	7.04	2.83	4.30	.00	1.78	1.35
3-1-05	32.40	.00	18.60	4.98	21.00	.00	12.45	3.77	11.30	.00	7.44	3.41	4.50	.00	1.88	1.51
3-1-06	42.30	.00	19.50	5.04	25.00	.00	13.00	4.27	12.30	.00	6.72	3.00	4.10	.00	1.82	1.42
MEAN	38.70	.00	18.65	4.94	22.26	.00	12.10	3.83	11.56	.00	7.72	3.25	4.30	.00	1.84	1.14
STAN DEV	4.13	.00	.70	.22	2.11	.00	.90	.24	.84	.00	1.01	.41	.16	.00	.05	.64

COMPOSITION C-4 (C-4) FREE AIR DETONATION

WEIGHT 100.00 GRAMS

CHANNEL	1		2		3		4		5		6		7		8	
Z VALUES	4.17		4.96		5.66		6.85		7.60		8.97		13.76		23.06	
	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP
50-0-20	39.60	.00	20.90	6.88	24.80	.00	16.80	5.33	15.90	.00	10.94	4.32	5.43	.00	4.22	2.65
50-0-21	60.90	.00	30.80	6.83	28.60	.00	18.30	5.62	15.30	.00	10.63	4.59	5.90	.00	3.95	2.33
50-0-22	46.20	.00	27.80	7.69			18.40	5.63	14.00	.00	10.14	3.83	4.95	.00	3.82	2.28
50-0-23	55.00	.00	29.10	6.66	26.40	.00	15.80	5.26	14.50	.00	11.04	4.34	6.10	.00	4.12	2.45
50-0-24	52.80	.00	24.85	5.97	20.00	.00	13.20	4.61	11.30	.00	7.84	2.99	4.60	.00	3.32	1.87
MEAN	50.72	.00	28.29	6.81	24.95	.00	16.50	5.29	14.20	.00	10.09	4.01	5.40	.00	3.87	2.32
STAN DEV	7.95	.00	2.20	.61	3.65	.00	2.14	.41	1.70	.00	1.32	.64	.63	.00	.35	.29

COMPOSITION C-4 (C-4) FREE AIR DETONATION

WEIGHT 125.00 GRAMS

CHANNEL	1		2		3		4		5		6		7		8	
Z VALUES	3.87		4.61		5.25		6.36		7.13		8.32		12.78		21.41	
	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP
50-0-15	76.80	.00	37.40	7.59					17.50	.00	13.24	4.58	5.60	.00	5.38	3.20
50-0-16	81.00	.00	33.30	7.27			22.30	6.43	16.00	.00	11.22	4.34	6.10	.00	6.02	3.30
50-0-17	87.10	.00	38.70	7.80	28.00	.00	20.30	5.92	14.85	.00	12.43	4.17	5.40	.00	5.12	2.97
50-0-18			36.10	7.17	24.00	.00	21.00	6.48	15.75	.00	12.94	4.76			5.45	3.38
50-0-19	73.45	.00	34.65	7.70	28.80	.00	21.80	6.74	21.80	.00	13.54	4.62	5.61	.00	5.35	4.42
MEAN	79.59	.00	36.03	7.51	27.47	.00	21.35	6.39	17.10	.00	12.64	4.50	5.68	.00	5.46	3.45
STAN DEV	5.00	.00	2.14	.27	2.31	.00	.88	.35	2.75	.00	.94	.24	.30	.00	.33	.56

Figure 4-29. Computer Print-out of Overpressure and Impulse - C-4 Free Air HE Equivalency Tests

COMPOSITION C-4 (C-4) CONFINED DETONATION

WEIGHT 50.00 GRAMS

CHANNEL	1		2		3		4		5		6		7		8	
Z VALUES	5.25		6.25		7.13		8.63		9.67		11.29		17.34		29.85	
	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP
4-1-07			10.50	2.52	10.56	.00	6.30	1.95	5.60	.00	3.00	1.49	2.40	.00	1.00	.00
4-1-08	22.40	.00	10.50	2.90	10.05	.00	5.60	1.84	7.00	.00	4.05	1.47	2.64	.00	1.22	.00
4-1-09	18.90	.00	10.00	2.23	10.20	.00	7.10	2.20	6.00	.00	4.05	1.10	1.90	.00	.72	.00
4-1-10	27.30	.00	12.00	2.79	9.30	.00	5.10	1.69	5.00	.00	3.15	1.14	2.80	.00	1.42	.00
4-1-11	18.90	.00	8.50	2.20	9.75	.00	6.00	1.92	6.00	.00	4.20	1.44	2.40	.00	.82	.00
MEAN	21.48	.00	10.30	2.46	9.97	.00	6.02	1.94	5.92	.00	3.85	1.35	2.46	.00	1.02	.00
STAN DEV	3.98	.00	1.25	.25	.48	.00	.75	.22	.73	.00	.42	.17	.35	.00	.28	.00

COMPOSITION C-4 (C-4) CONFINED DETONATION

WEIGHT 75.00 GRAMS

CHANNEL	1		2		3		4		5		6		7		8	
Z VALUES	4.59		5.46		6.23		7.54		8.45		9.87		15.15		25.38	
	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP
52-0-01			10.20	3.05			7.60	2.46			6.50	2.48			1.32	.00
52-0-02	28.60	.00	10.00	2.95	14.25	.00	7.50	2.46			6.20	2.23	2.80	.00	1.72	.00
52-0-03	26.40	.00	12.30	2.71	15.00	.00	8.00	2.40			5.30	1.89	2.90	.00	1.32	.00
52-0-04	27.50	.00	10.50	2.64	13.75	.00	7.20	2.22	6.60	.00	5.90	2.27	3.00	.00	1.42	.00
52-0-05	26.40	.00	11.40	3.19	10.50	.00	7.20	2.74	6.50	.00	6.20	2.47	3.20	.00	1.50	.00
52-0-06			12.90	3.07	16.30	.00	7.60	2.44	6.90	.00	6.10	2.26	3.20	.00	1.76	.00
MEAN	27.22	.00	11.22	2.94	13.96	.00	7.52	2.47	6.67	.00	6.03	2.27	3.02	.00	1.49	.00
STAN DEV	1.05	.00	1.19	.22	2.16	.00	.30	.17	.21	.00	.41	.21	.10	.00	.22	.00

COMPOSITION C-4 (C-4) CONFINED DETONATION

WEIGHT 100.00 GRAMS

CHANNEL	1		2		3		4		5		6		7		8	
Z VALUES	4.17		4.96		5.66		6.85		7.68		8.97		13.76		23.06	
	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP
50-0-35	26.40	.00	12.60	3.40	15.20	.00	9.20	3.10	10.00	.00	6.20	2.54	4.30	.00	2.62	1.40
50-0-36	33.00	.00	12.60	3.40	13.60	.00	8.40	3.22	8.00	.00	5.80	2.43	3.40	.00	3.02	1.27
11-1-16	33.90	.00	19.35	4.94	16.80	.00	10.60	4.64	8.80	.00	7.80	3.41	3.90	.00	2.12	1.56
11-1-17	22.10	.00	16.65	4.79	12.60	.00	11.20	3.94	8.20	.00	6.30	5.65	3.20	.00	2.34	1.65
11-1-18	32.50	.00	21.05	6.49	19.20	.00	13.00	5.02	9.20	.00	7.50	3.83	4.00	.00		
MEAN	29.56	.00	16.45	4.62	15.48	.00	10.48	4.00	8.84	.00	6.56	3.57	3.76	.00	2.51	1.48
STAN DEV	5.10	.00	3.85	1.27	2.62	.00	1.79	.82	.80	.00	.60	1.30	.45	.00	.39	.17

COMPOSITION C-4 (C-4) CONFINED DETONATION

WEIGHT 125.00 GRAMS

CHANNEL	1		2		3		4		5		6		7		8	
Z VALUES	3.87		4.61		5.25		6.36		7.13		8.32		12.78		21.41	
	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP	PSI	IMP
51-0-01			16.20	3.39	18.40	.00	12.80	4.31	10.00	.00	7.50	3.22	5.80	.00	5.90	.00
51-0-02	45.50	.00	19.00	4.66	16.00	.00			10.00	.00	6.20	2.82	6.00	.00		
51-0-03	40.30	.00	20.40	4.70	20.00	.00	12.00	4.23	10.00	.00	6.60	2.81	7.40	.00	4.10	1.36
51-0-04	52.60	.00	18.00	4.74	17.00	.00	11.20	3.85	9.90	.00	7.80	3.01	6.60	.00	3.90	.00
51-0-05			17.10	3.99	13.60	.00			9.50	.00	7.50	2.67			3.42	.00
51-0-06	24.70	.00	15.30	4.43					10.00	.00	6.90	2.77	5.80	.00	3.70	.00
51-0-07	39.00	.00	17.10	4.63	14.40	.00	13.80	4.19	10.00	.00	7.20	2.74	5.00	.00	4.30	.00
MEAN	40.30	.00	17.70	4.36	16.57	.00	12.45	4.14	9.91	.00	7.10	2.86	6.10	.00	4.22	.23
STAN DEV	10.11	.00	1.85	.50	2.41	.00	1.11	.20	.19	.00	.57	.19	.82	.00	.80	.56

Figure 4-30. Computer Print-out of Overpressure and Impulse - C-4 Confined HE Equivalency Tests

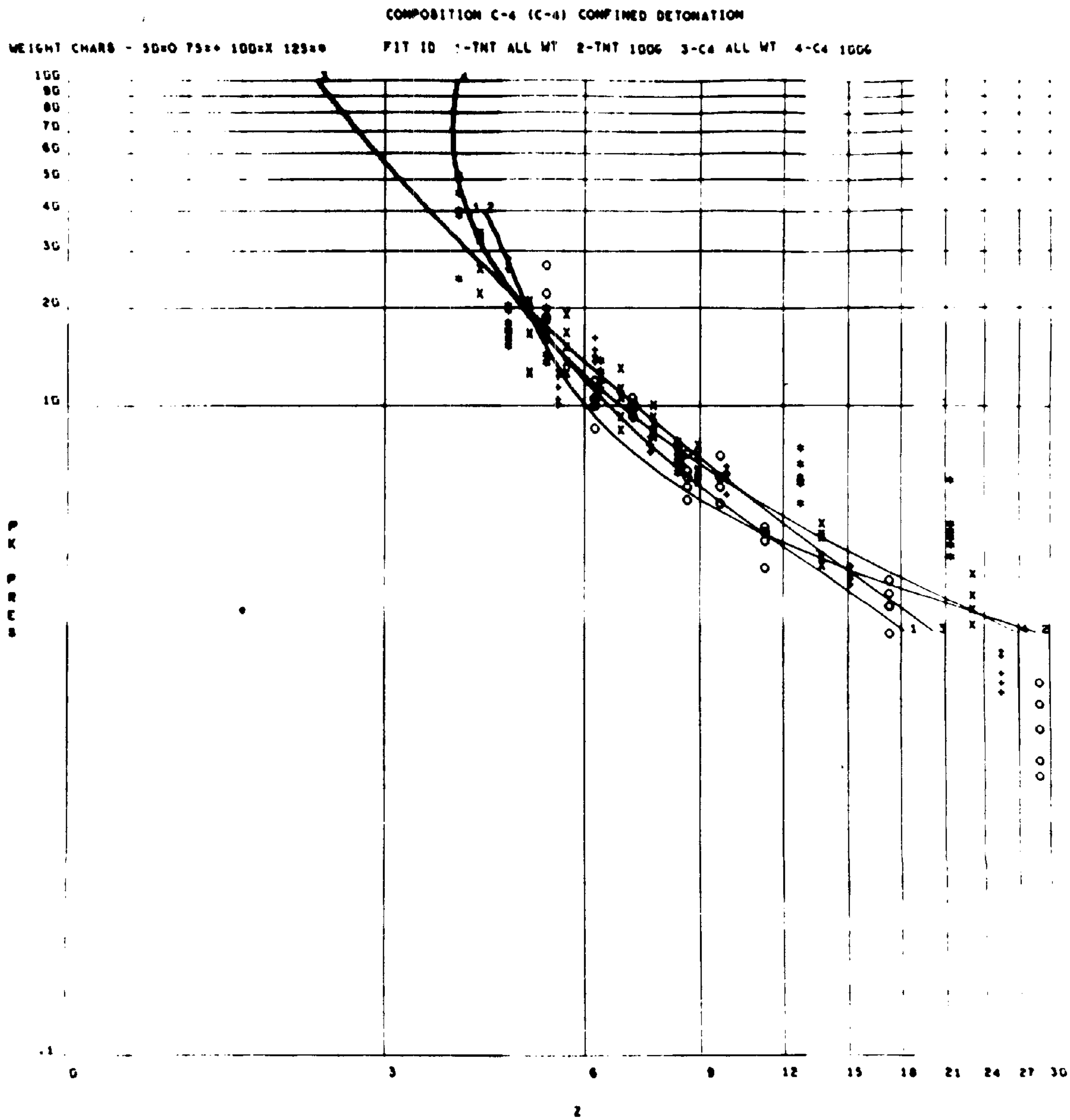


Figure 4-31. Summary - C-4 Confined with Reference Curves

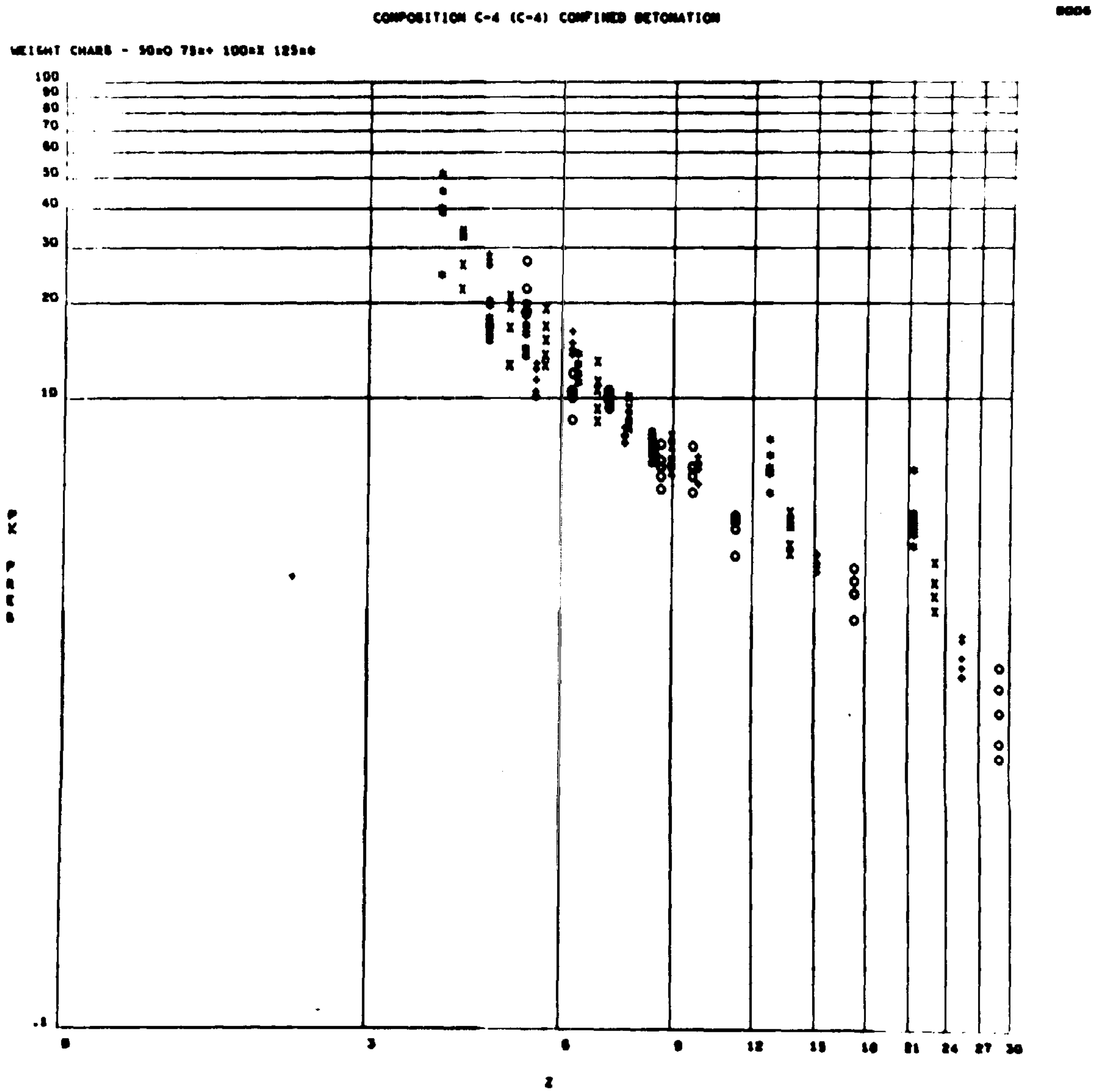


Figure 4-32. C-4 Confined Reference Data Points

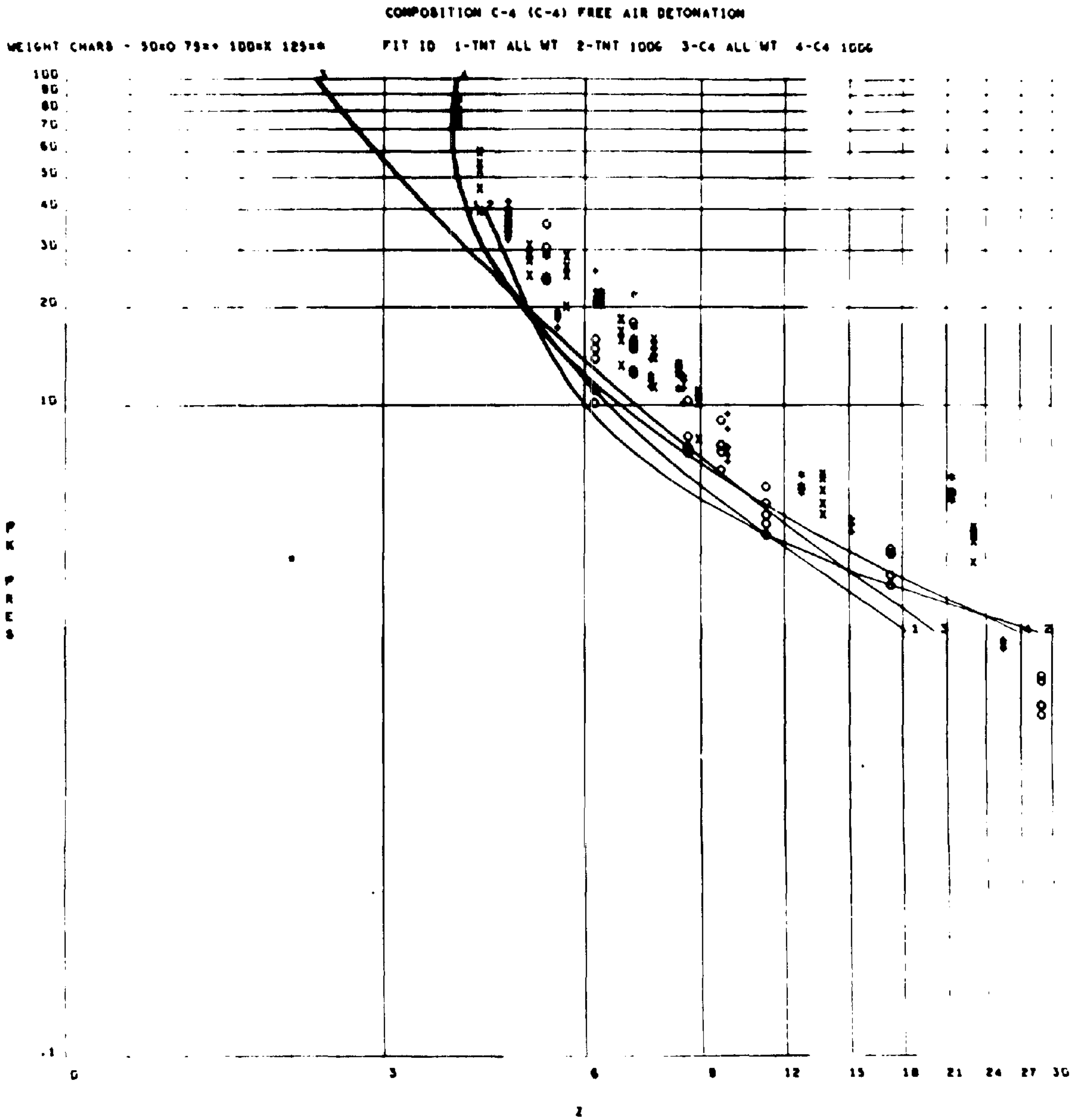


Figure 4-33. Summary - C-4 Free Air with Reference Curves

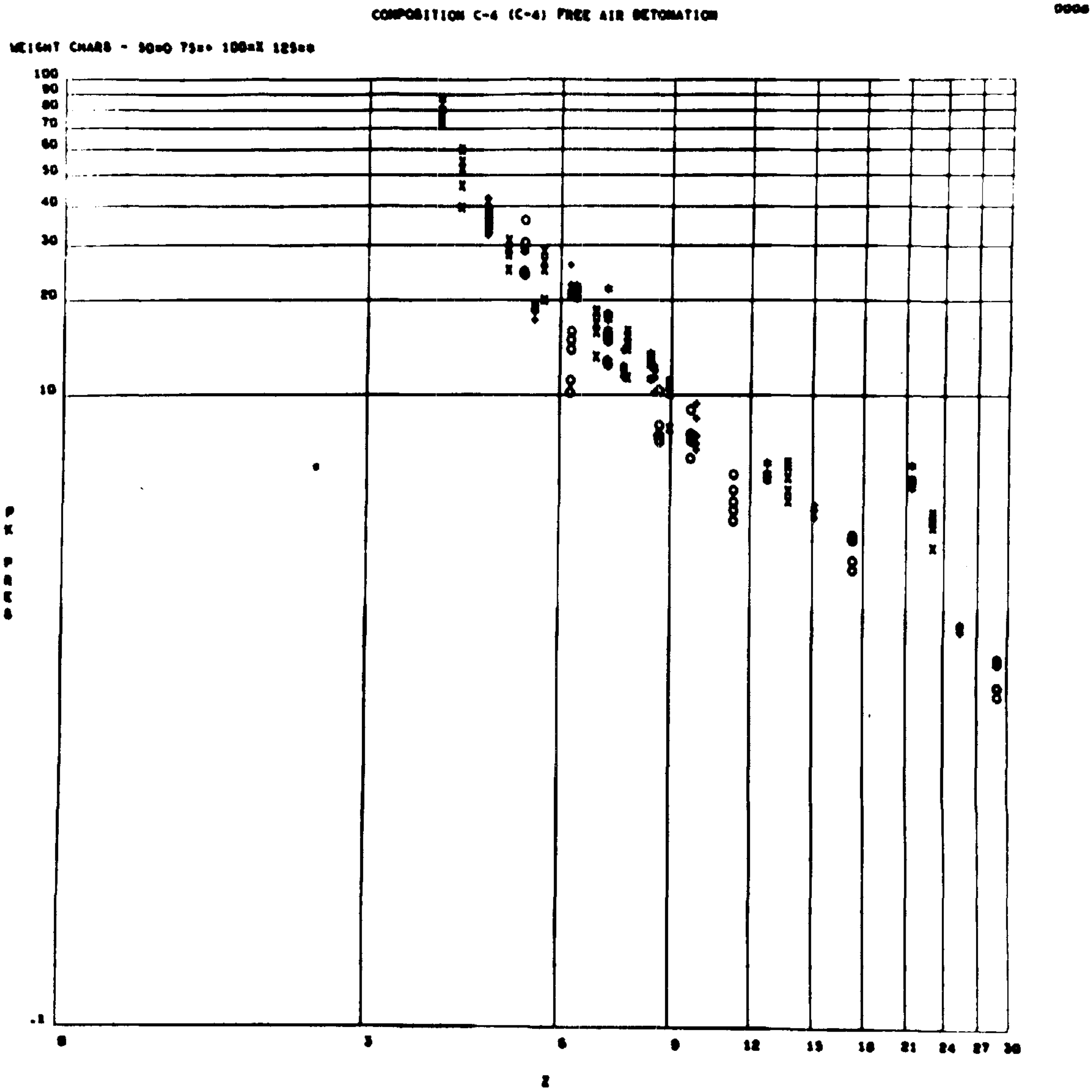


Figure 4-34. C-4 Free Air Reference Data Points

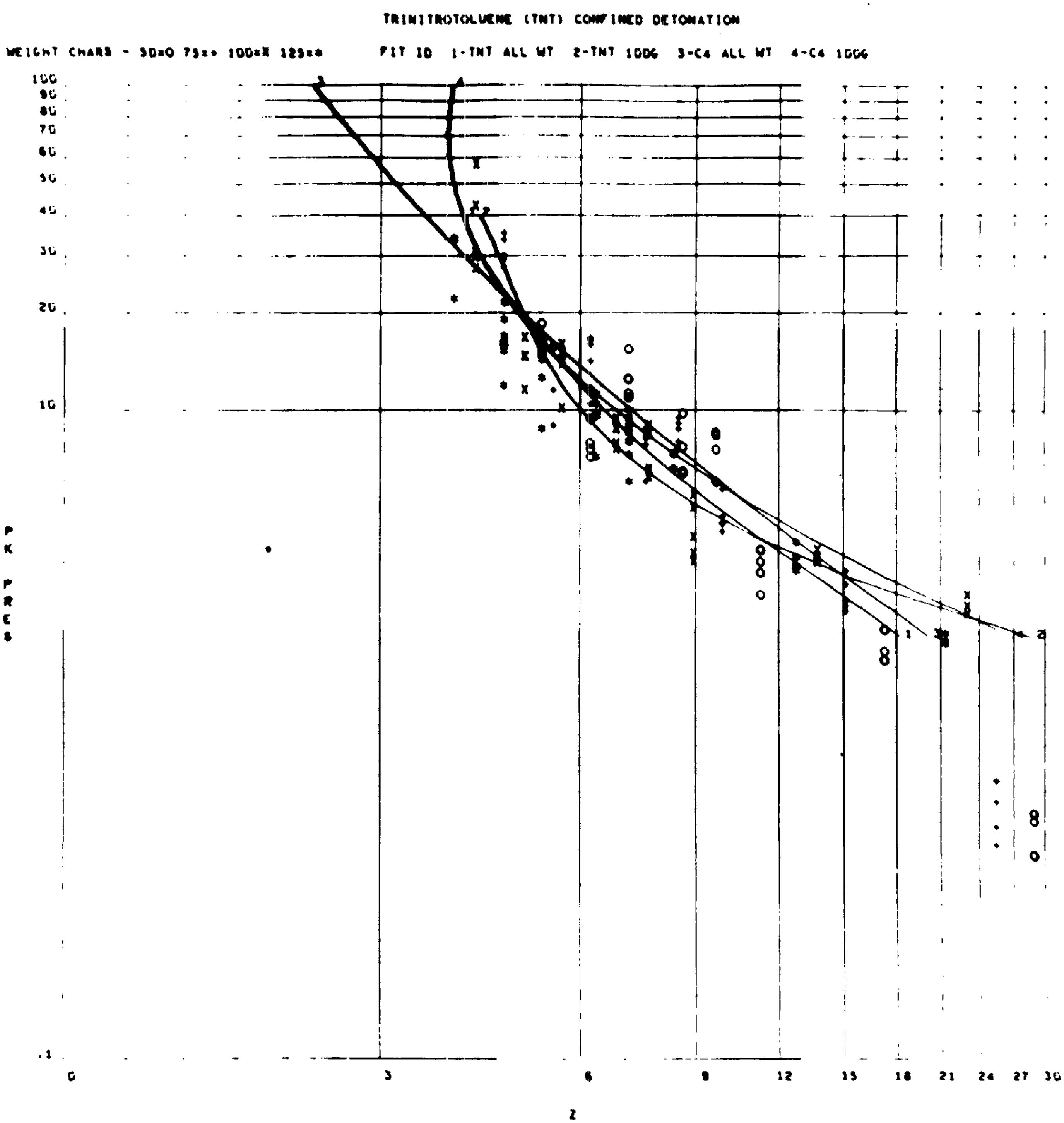


Figure 4-35. Summary - TNT Confined with Reference Curves

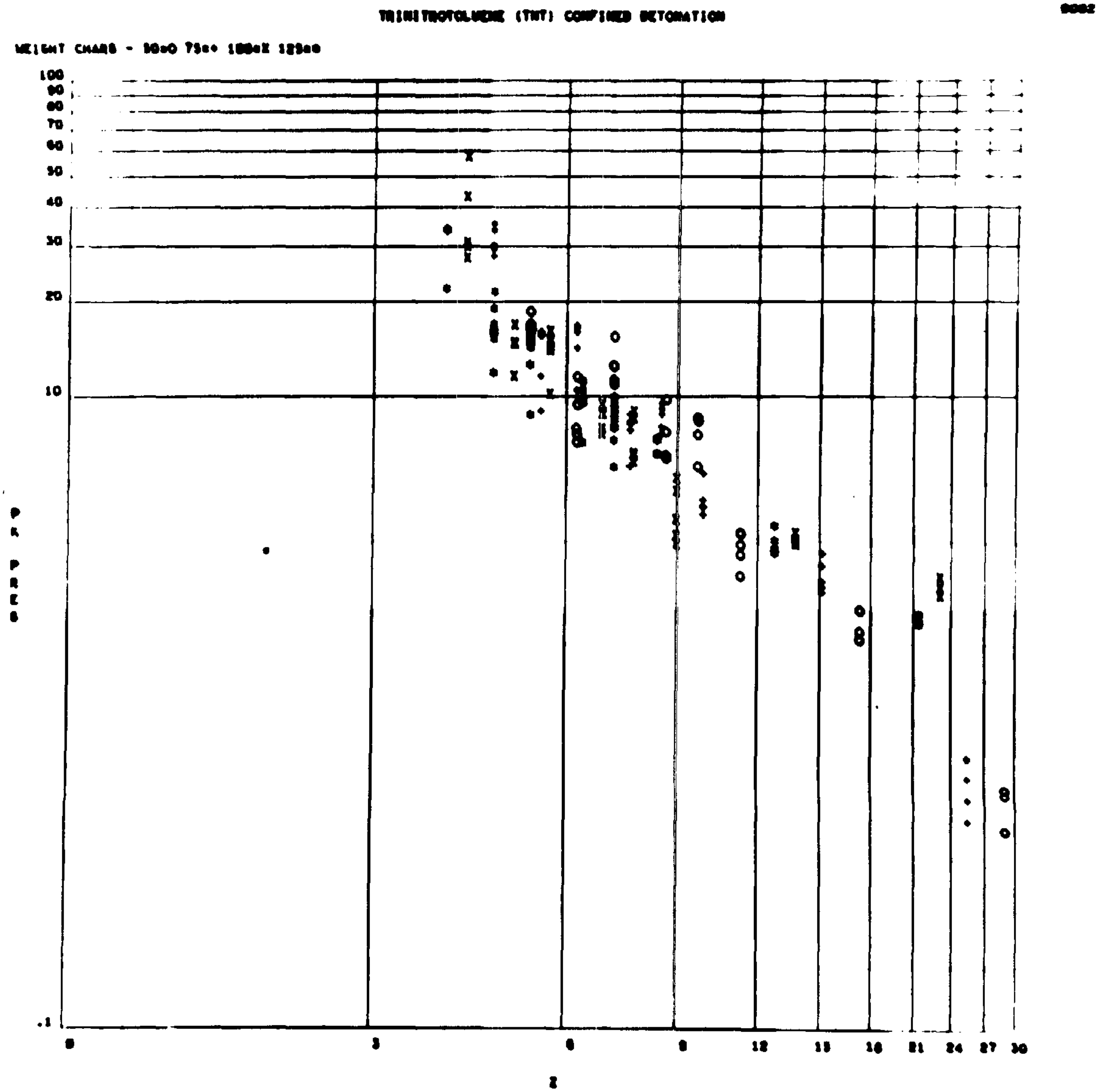


Figure 4-36. TNT Confined Reference Data Points

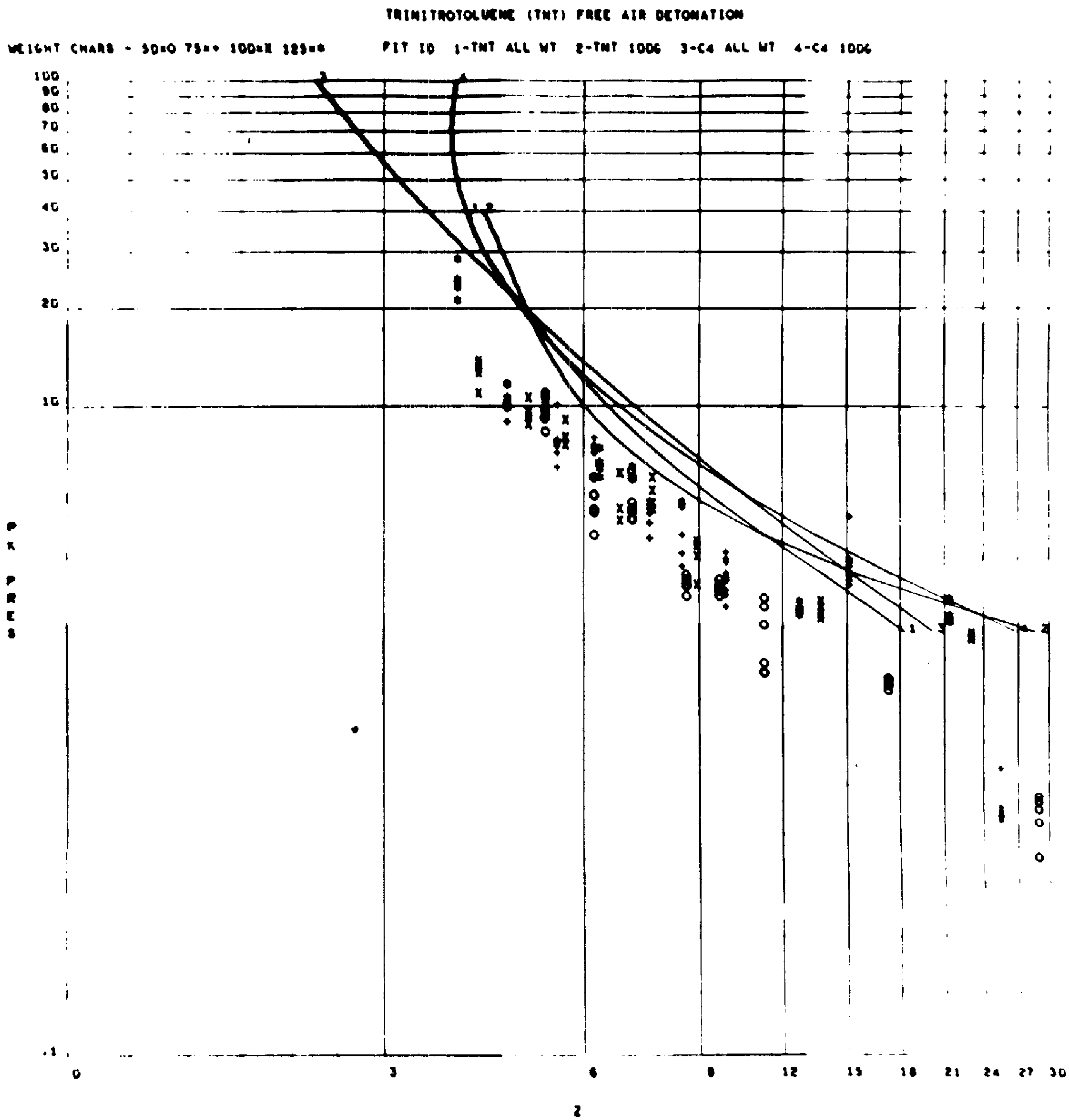


Figure 4-37. Summary - TNT Free Air with Reference Curves

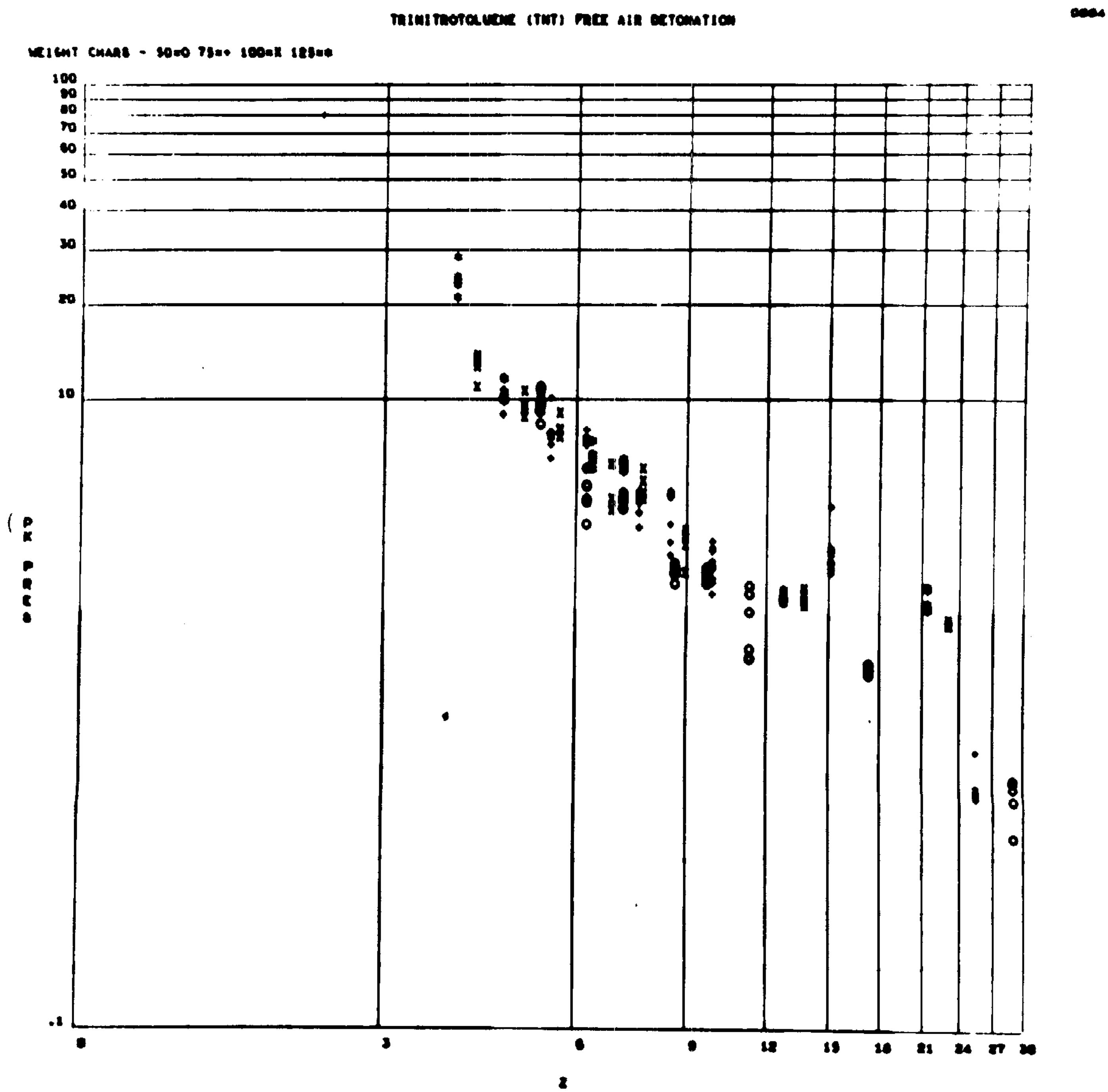


Figure 4-38. TNT Free Air Reference Data Points

4.4.4.2 Impulse

Although not used in the calculations of HE equivalency, impulse data for the high explosives was derived and is shown in the digital tab runs, Figures 4-27 through 4-30. Scatter diagrams of the impulse data for the high explosive calibration plots are shown in Figures 4-39 through 4-42. Because of the erratic values which resulted, no curve fitting routines were applied to the data. Impulse values for pyrotechnics were not derived because of the extremely erratic nature of the data and since most TNT equivalency comparisons are made on the basis of peak side-on overpressure.

Impulse data can be obtained if desired by the computer program currently available.

4.4.5 PYROTECHNIC HE EQUIVALENCIES

The capability of pyrotechnics to produce a characteristic pressure-time profile similar to that of a high explosive and which could be used to calculate a TNT equivalency value was undertaken in Phase I activities. Values for the various sulfur- and lactose-based pyrotechnics range from 3.7 to 7.1 percent TNT when compared to bare spherical pentolite. Confinement of the pyrotechnic material in the double capped steel tube used in this phase when compared with high explosives confined in an identical manner changed the TNT equivalency value of the material significantly. Utilizing the best fit calculation presented in Appendix E, characteristic overpressure versus scaled distance (Z) curves were developed for the TNT equivalency values computed. A plot of the various pyrotechnics tested and computer fitted curves of the data are shown in Figures 4-43 through 4-50.

Computation of equivalency values for each of the pyrotechnic materials was performed utilizing the 100 gram confined high explosives TNT and C-4 reference data. This approach compensated for any irregularities in sample configuration or geometry. The values shown in Table 4-5 show an average value as computed at several scaled distances (Z's) since the curves of the high explosives vary through the area of interest as shown in Figure 4-51. The curve for pyrotechnics is an average as calculated from the data shown in Appendix F.

The alternate method of calculating HE equivalencies is the direct ratio method which is shown graphically in Table 4-6. This method is valid only when identical material weights and distances are compared. It should be pointed out that this is not the generally accepted method for calculating TNT (or HE) equivalencies but is a quick and easy method for working a comparison within the parameters stated above.

It is to be noted that there is no HE equivalency value for the sample of Sulfur Yellow due to the failure of the material to detonate under test conditions. A review of Phase I final report shows that the value of 3.72 percent was obtained by increasing the sample temperature to 100°F prior to the test.

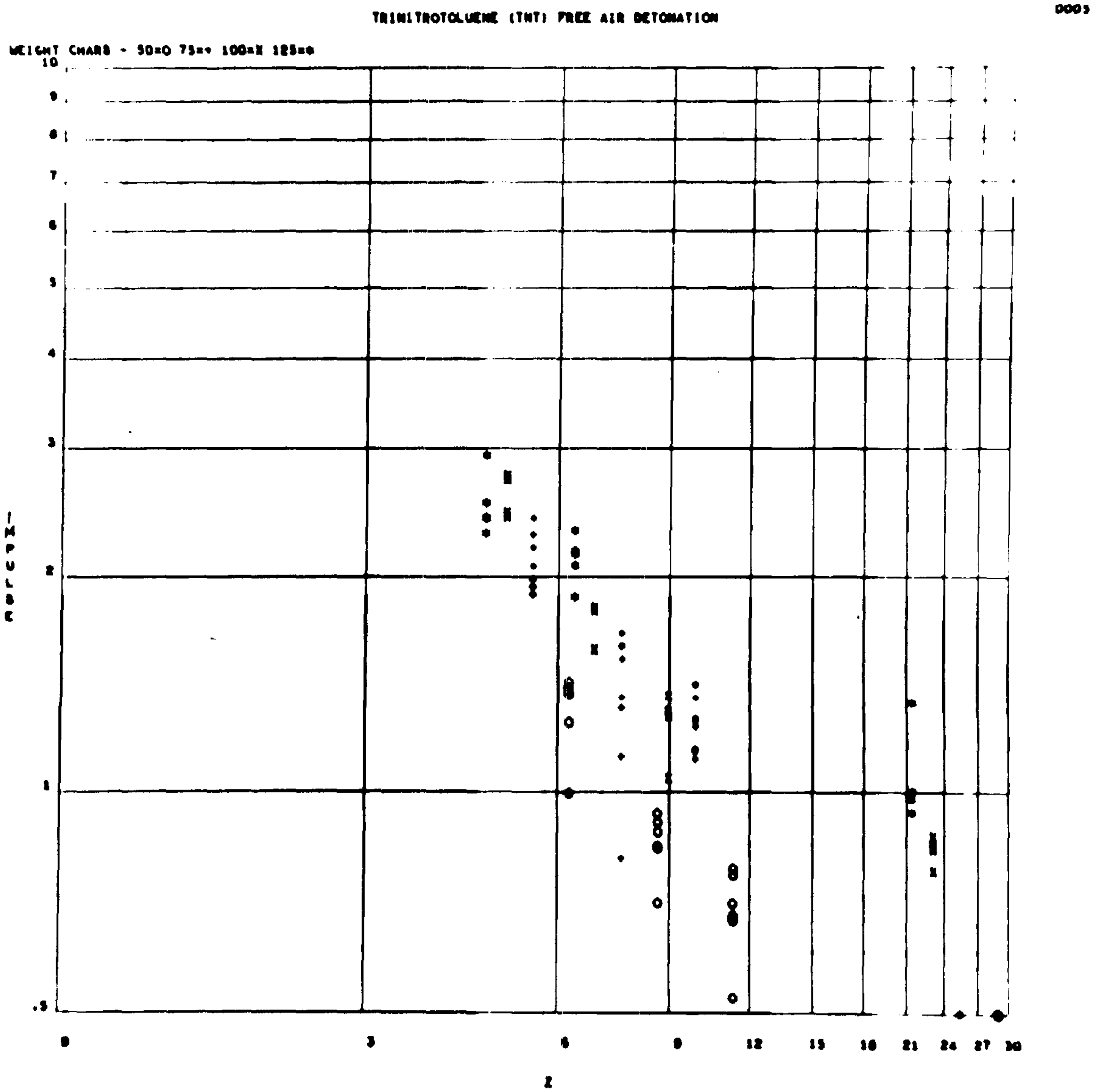


Figure 4-39. TNT Free Air Impulse Reference Data

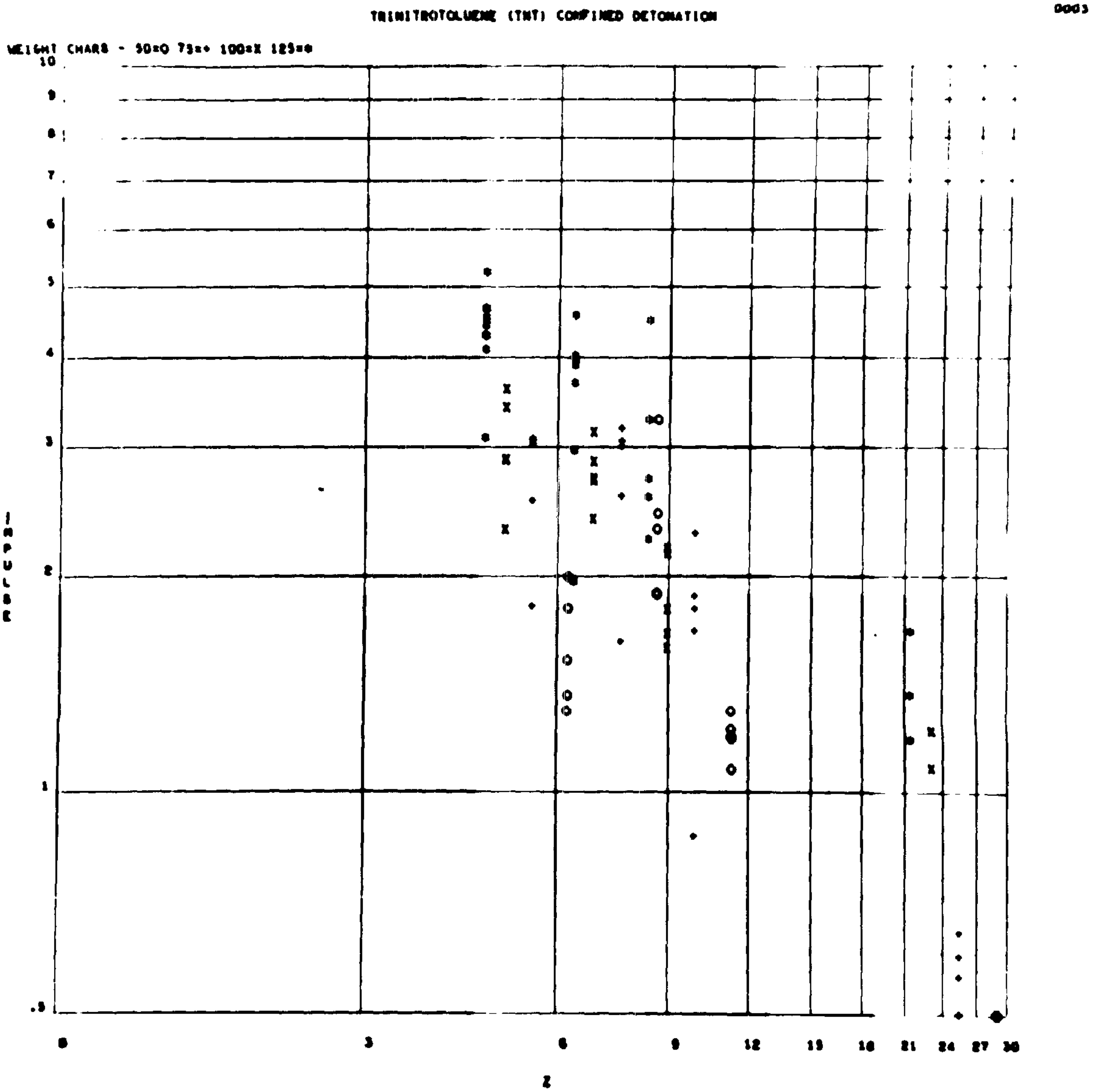


Figure 4-40. TNT Confined Impulse Reference Data

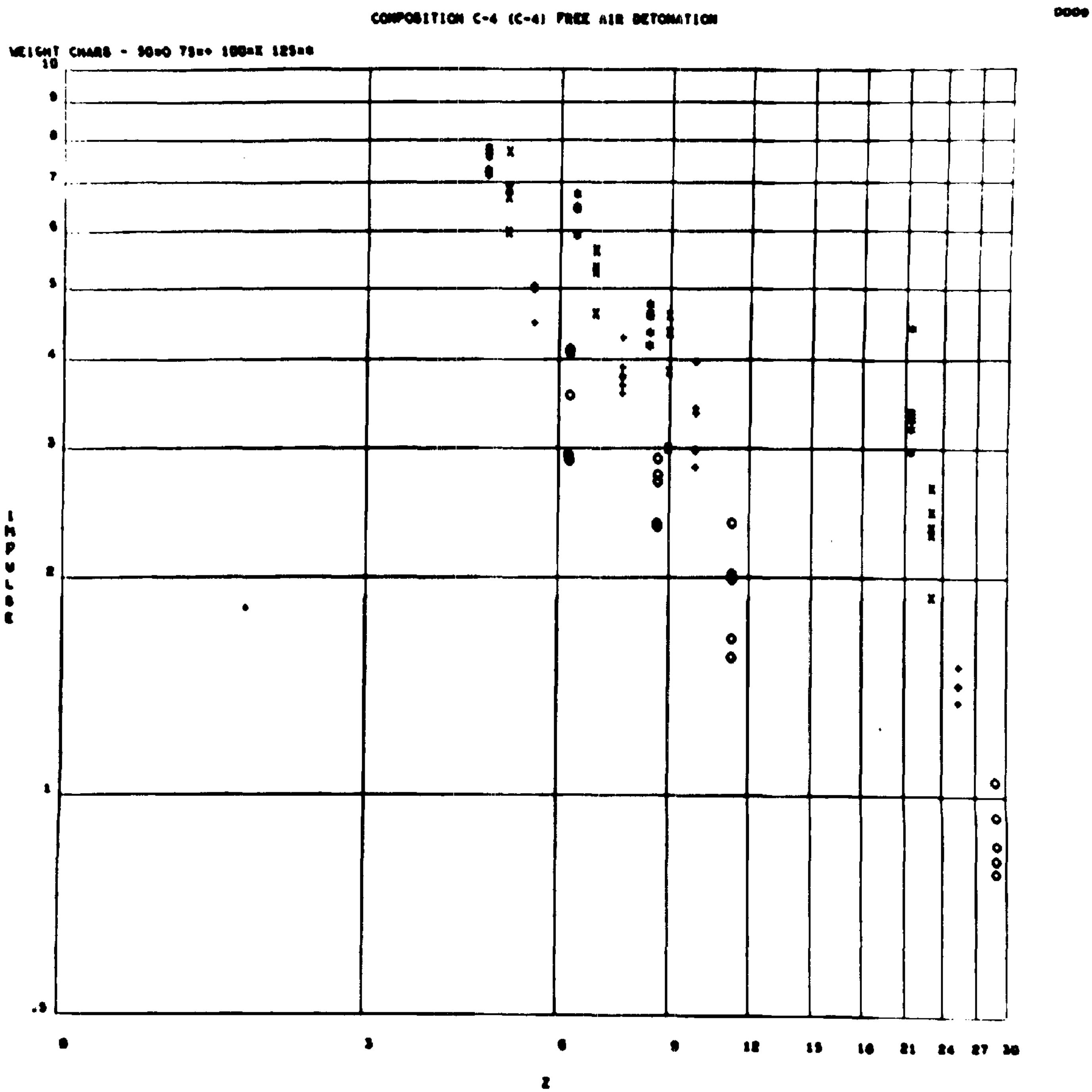


Figure 4-41. C-4 Free Air Impulse Reference Data

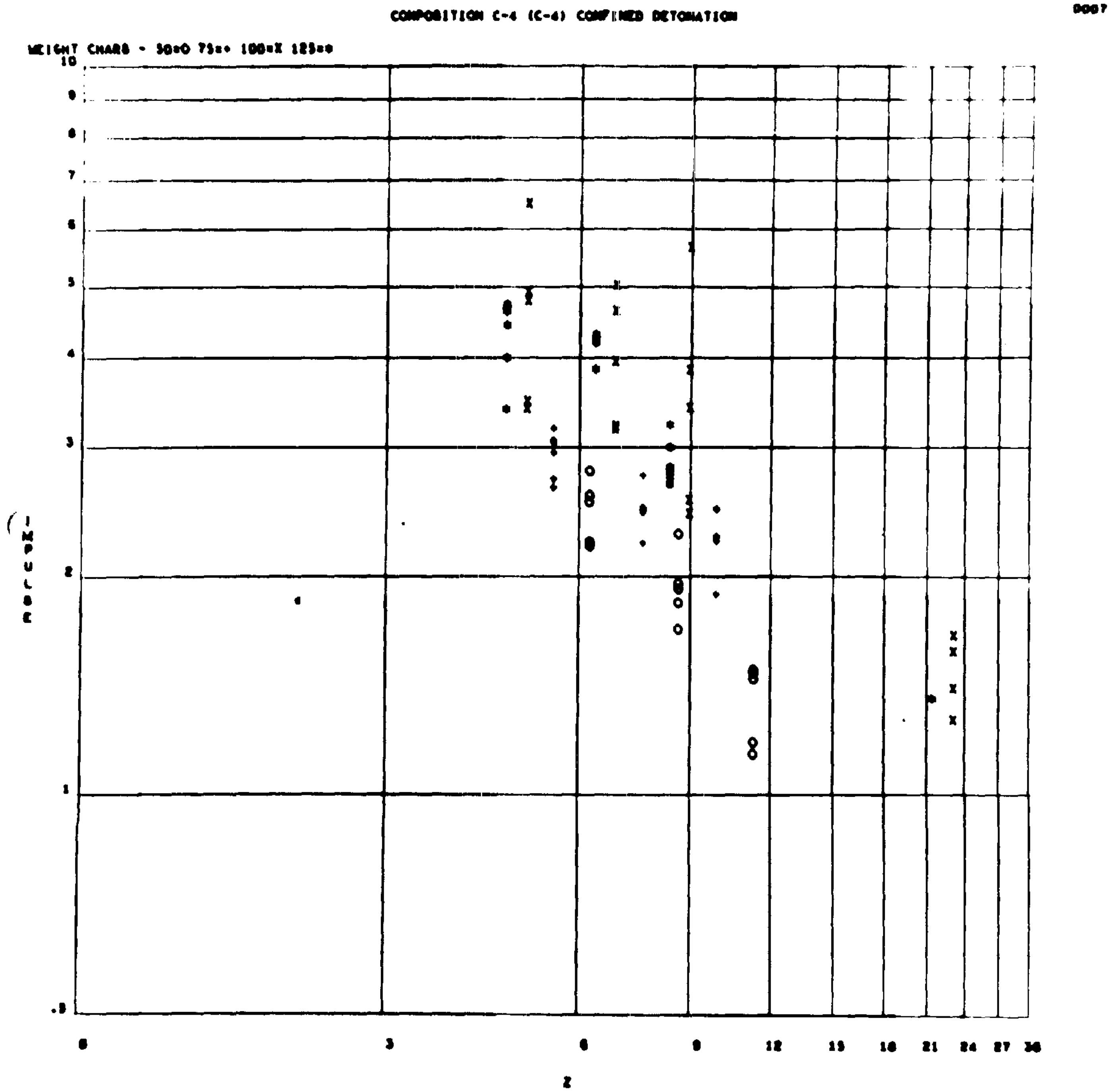


Figure 4-42. C-4 Confined Impulse Reference Data

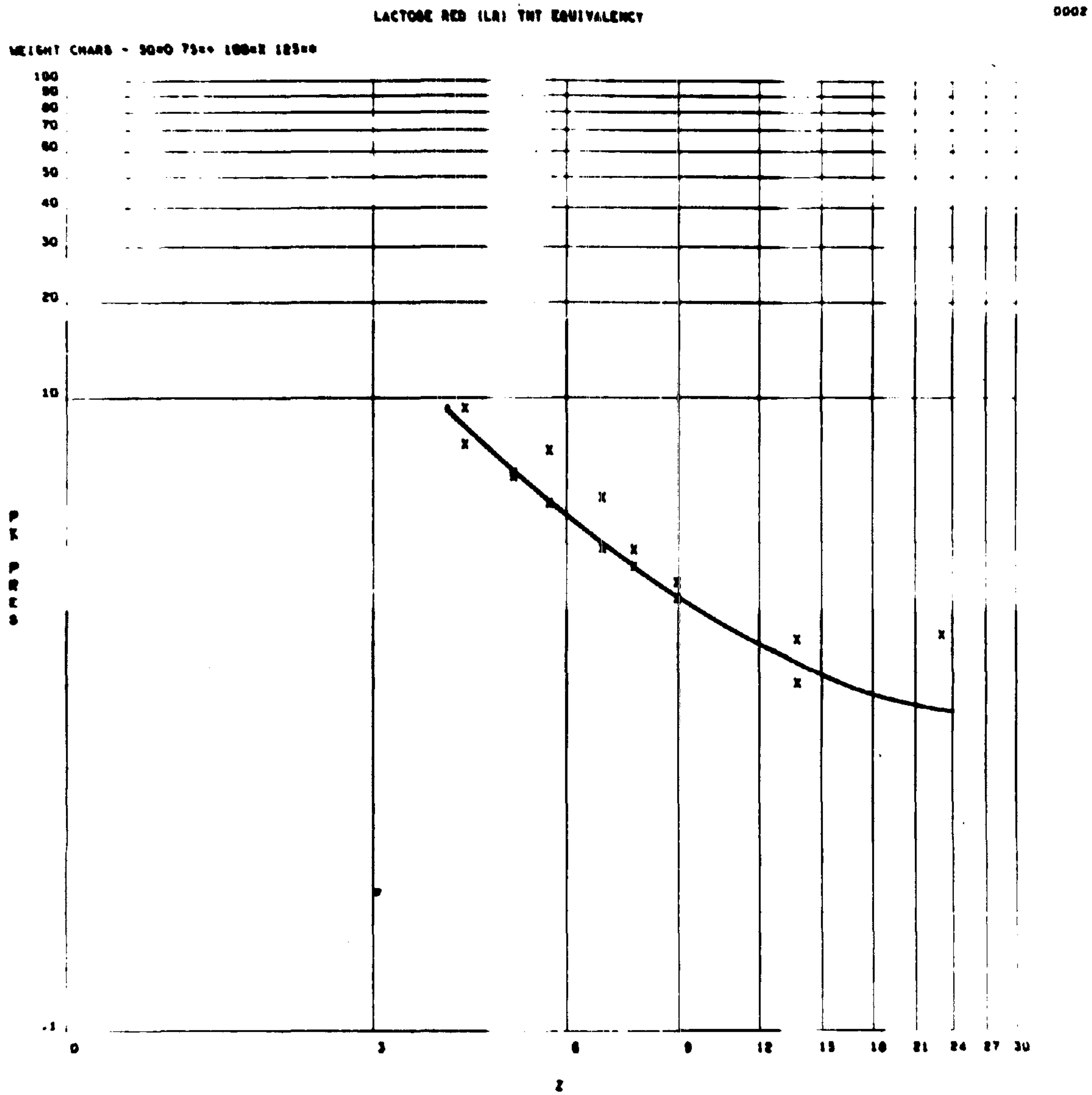


Figure 4-43. Pyrotechnic Lactose Red

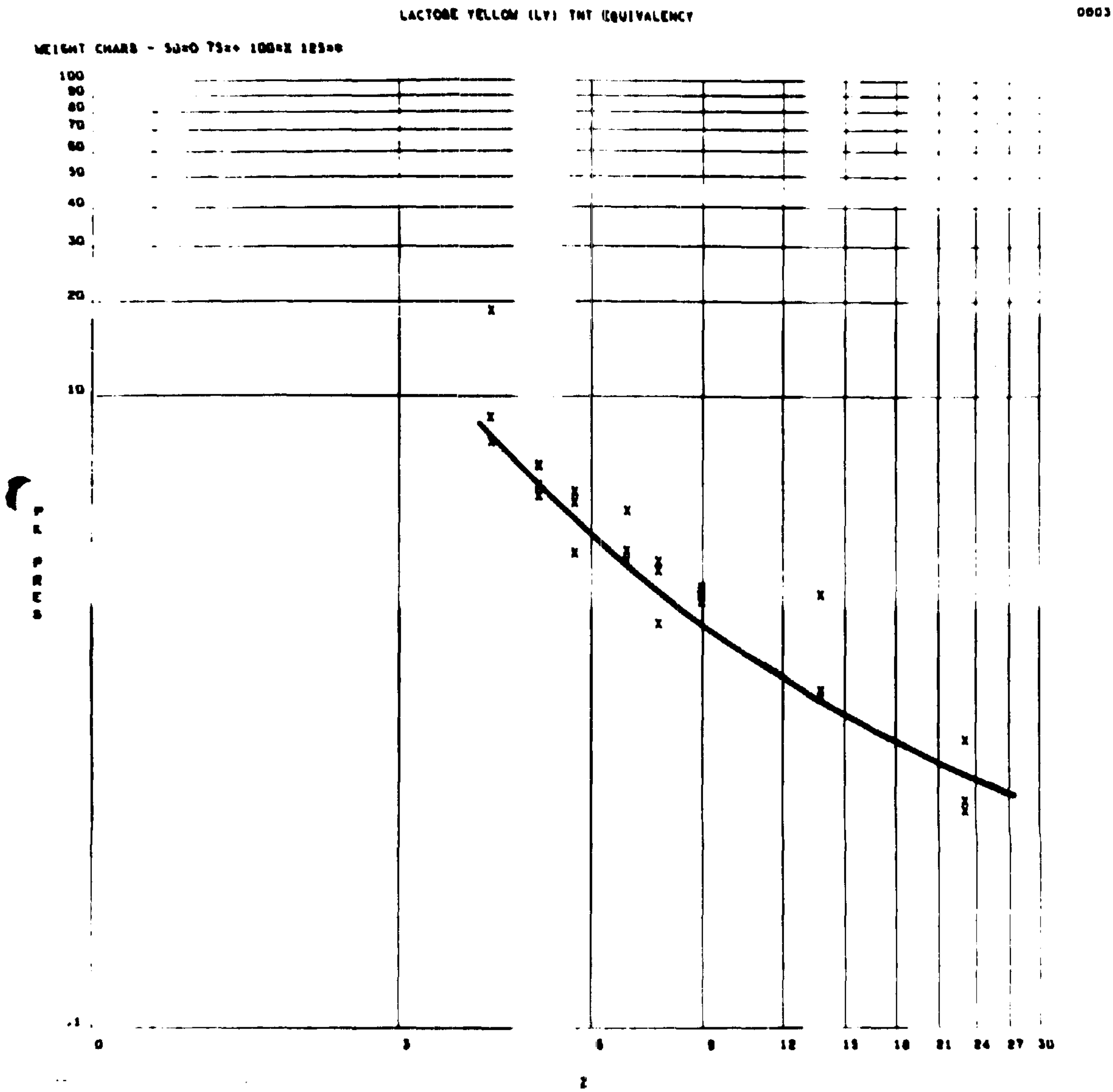


Figure 4-44. Pyrotechnic Lactose Yellow

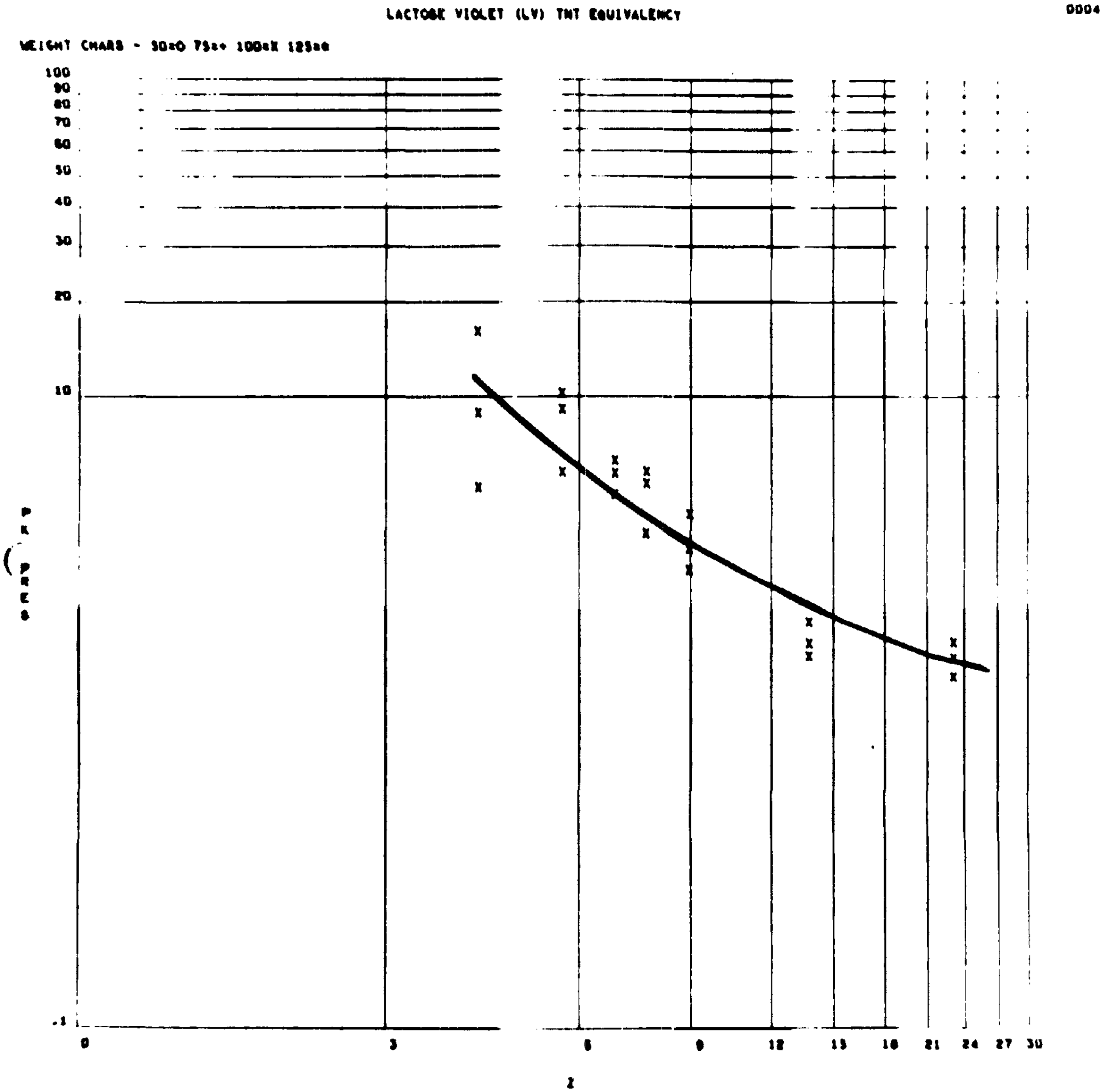


Figure 4-45. Pyrotechnic Lactose Violet

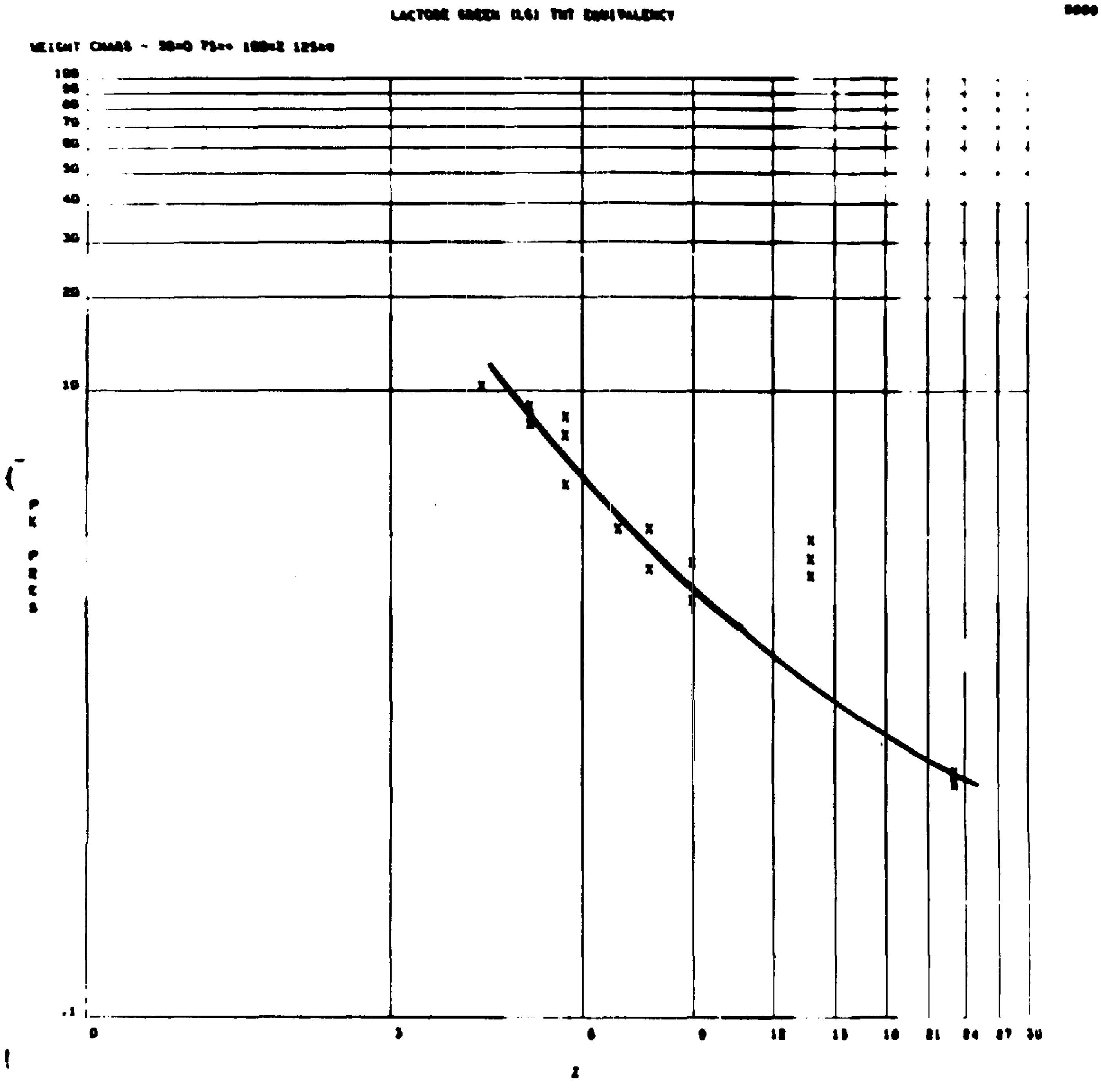


Figure 4-46. Pyrotechnic Lactose Green

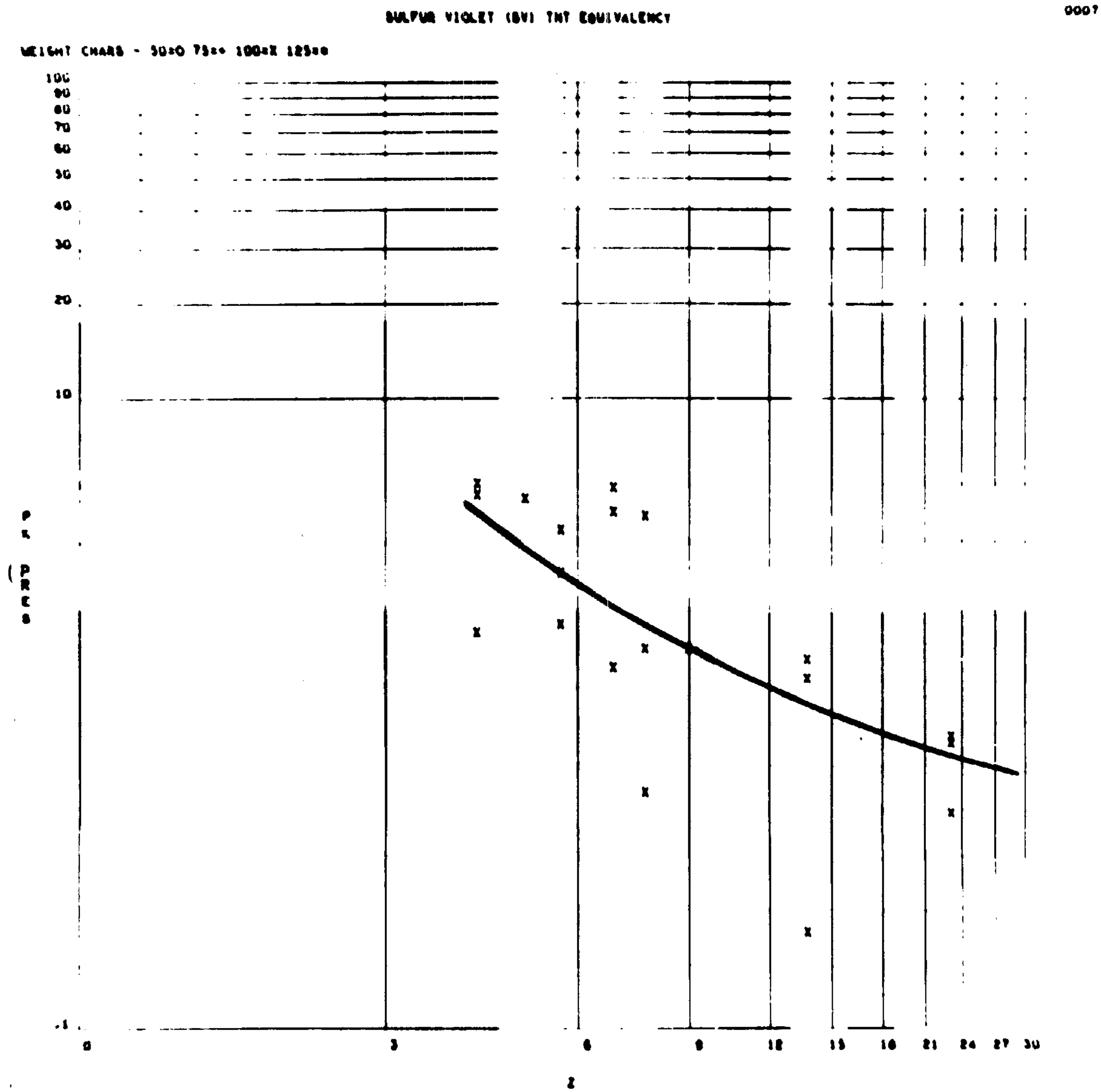


Figure 4-47. Pyrotechnic Sulfur Violet

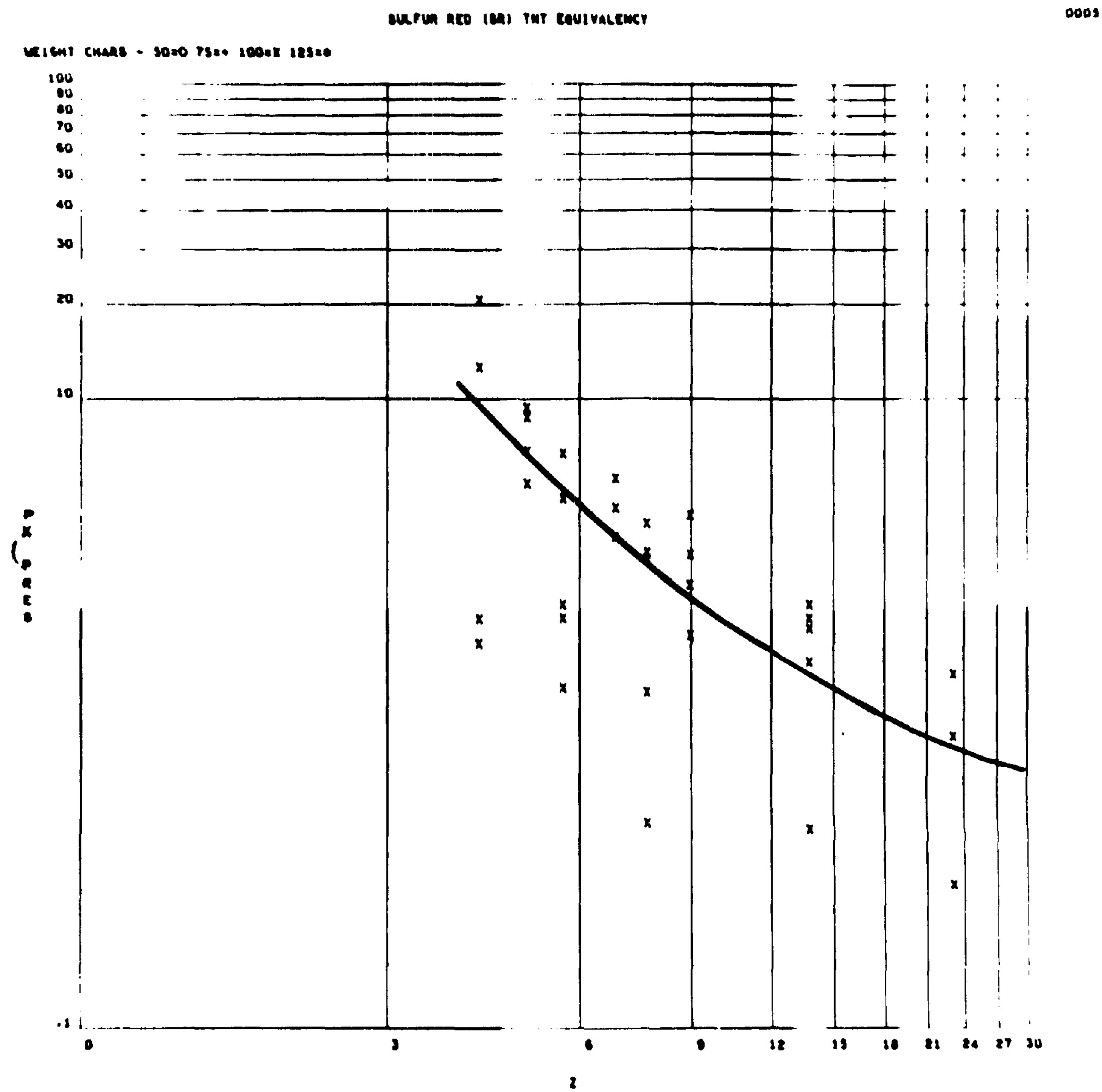


Figure 4-48. Pyrotechnic Sulfur Red

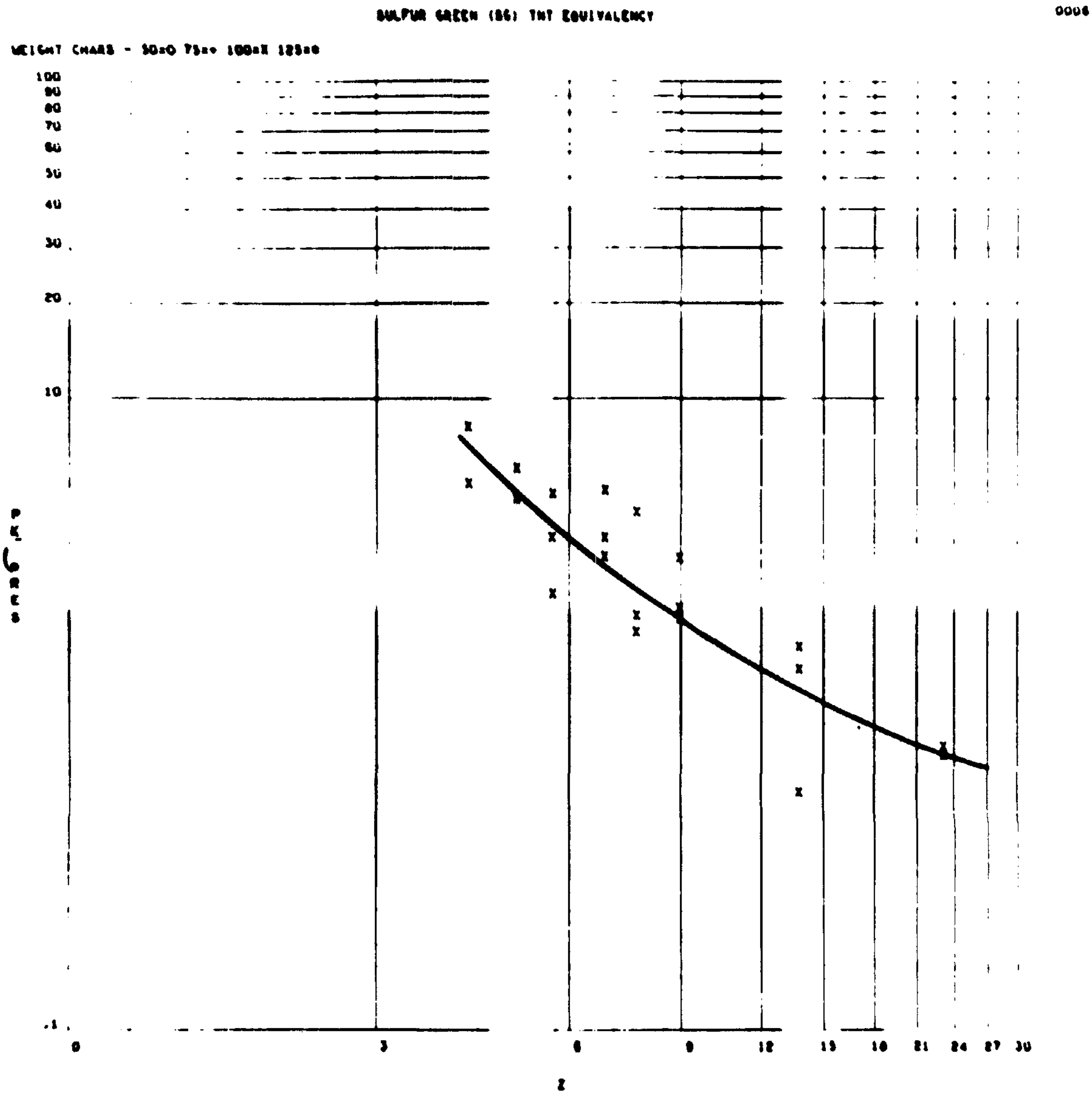


Figure 4-49. Pyrotechnic Sulfur Green

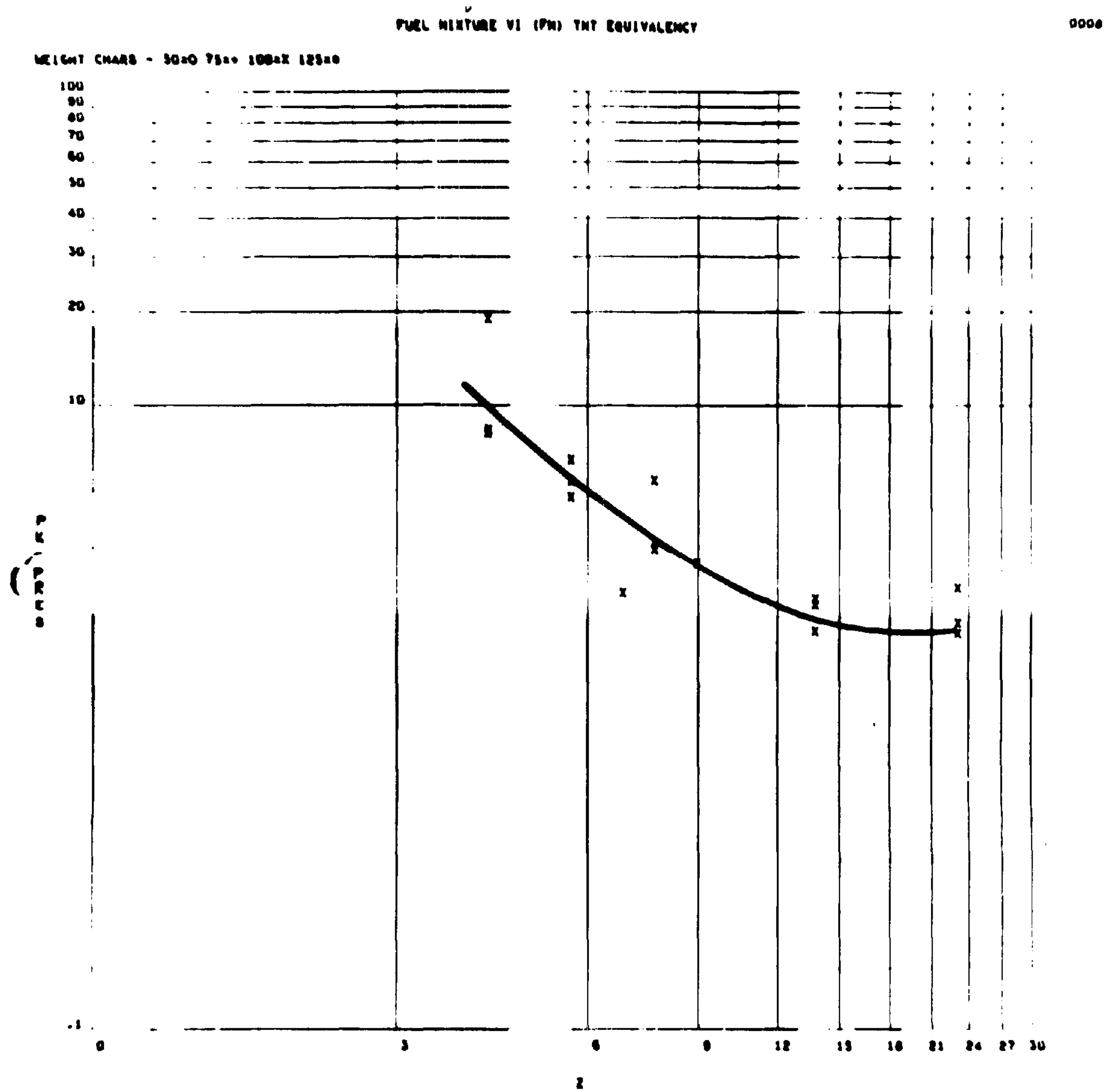


Figure 4-50. Pyrotechnic Fuel Mix

Table 4-5. Pyrotechnic TNT/C-4 Equivalency Values

SAMPLE CONFINED 100 GRAMS	% TNT - CONFINED TNT 100 GRAMS	%C-4 CONFINED C-4 100 GRAMS
Lactose Red	6.92	4.99
Lactose Yellow	4.88	3.58
Lactose Violet	13.29	9.44
Lactose Green	11.44	7.63
Sulfur Red	6.79	5.09
Sulfur Yellow	NONE	NONE
Sulfur Violet	2.64	1.74
Sulfur Green	4.12	2.72
Fuel Mixture	14.10	11.44

Table 4-6. Comparison of % TNT Values at $Z = 5.25$ Using the Direct Pressure Ratio Method

Material Tested	Peak Pressure	% TNT	Remarks (Source)
Pentolite	30.06	100.00	Soroka (Goodman)
Pentolite	28.60	95.00	Ø I
C-4	29.00	96.40	Ø I
TNT Flaked (Free Air)	8.60	28.70	Ø III
TNT Flaked (Confined)	14.25	46.50	Ø III
C-4 (Free Air)	27.00	90.00	Ø III
C-4 (Confined)	18.00	60.00	Ø III
LV (Confined)	@8.00	26.60	Ø III

The values shown above are computed using the technique discussed in Appendix E, Calculations and Data, wherein a new scaled distance Z_2 is calculated based on the actual pressure acquired from the test event for a particular pyrotechnic. Using the new scaled distance Z_2, the new W (weight of high explosive) is determined. The percent HE value is then the ratio of the new W (weight of HE) to the original weight of the sample tested.

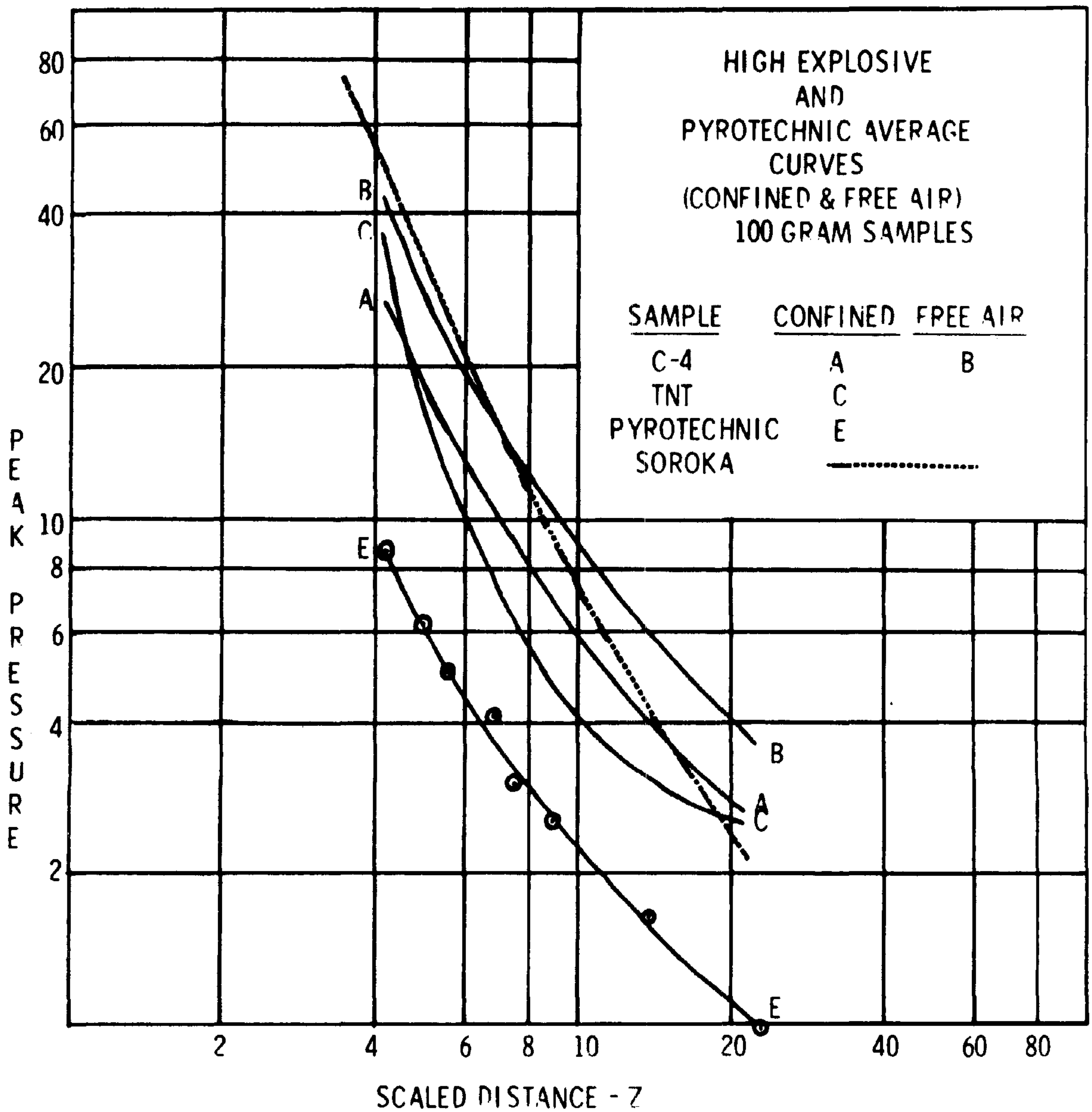


Figure 4-51. High Explosive and Pyrotechnic Average Curves (Confined & Free Air) 100 Gram Samples

4.4.6 FRAGMENTATION STUDY

4.4.6.1 General

The factors involved in establishing the characteristics of HE equivalency testing include:

- Pyrotechnic Sample Mass
- HE standard Mass
- Container Geometry
- Container rupture strength

These parameters influence the observable effects, which include:

- Blast wave characteristics
- Fragment quantity and mass distribution
- Fragment velocity

The blast wave characteristics were studied in detail and reported in previous paragraphs of this section. This paragraph is concerned with the capability to classify reactions based on fragmentation characteristics. Its purpose is to determine the following:

- Fragmentation characteristics directly related to high explosives at varying weights with the geometry of the container fixed.
- Comparison of fragmentation characteristics of high explosives and pyrotechnics.
- Contribution of container parameters (wall thickness versus sample weight).
- Fragmentation velocities of high explosives versus pyrotechnics.

4.4.6.2 High Explosive Fragments

Flaked TNT and C-4 were tested in the standard TNT equivalency container (Figure 4-52). The two samples were tested at varying weights of 50, 75, 100 grams. The results were similar for the two samples. At 50 grams, both the C-4 and flaked TNT cut a hole in the bottom cup similar to an explosion as defined by the card gap test (Figure 4-53). The 75-gram sample had a slightly more violent reaction, cutting a hole in both the top and bottom caps of the container. In one case, the 75-gram C-4 split the pipe as well (Figure 4-54). The 100-gram samples created multiple fragmentation (Figure 4-55). The reaction effects on the vessel were similar for C-4 and flaked TNT.

Test results were consistent with the interpretation that the C-4 and flaked TNT reactions were identical, with High Velocity Detonations (HVD) having occurred in all of the 50, 75, and 100 gram tests. It is therefore postulated that the quantity and size of the fragments are effectively dependent only upon the mass of explosive in the container.

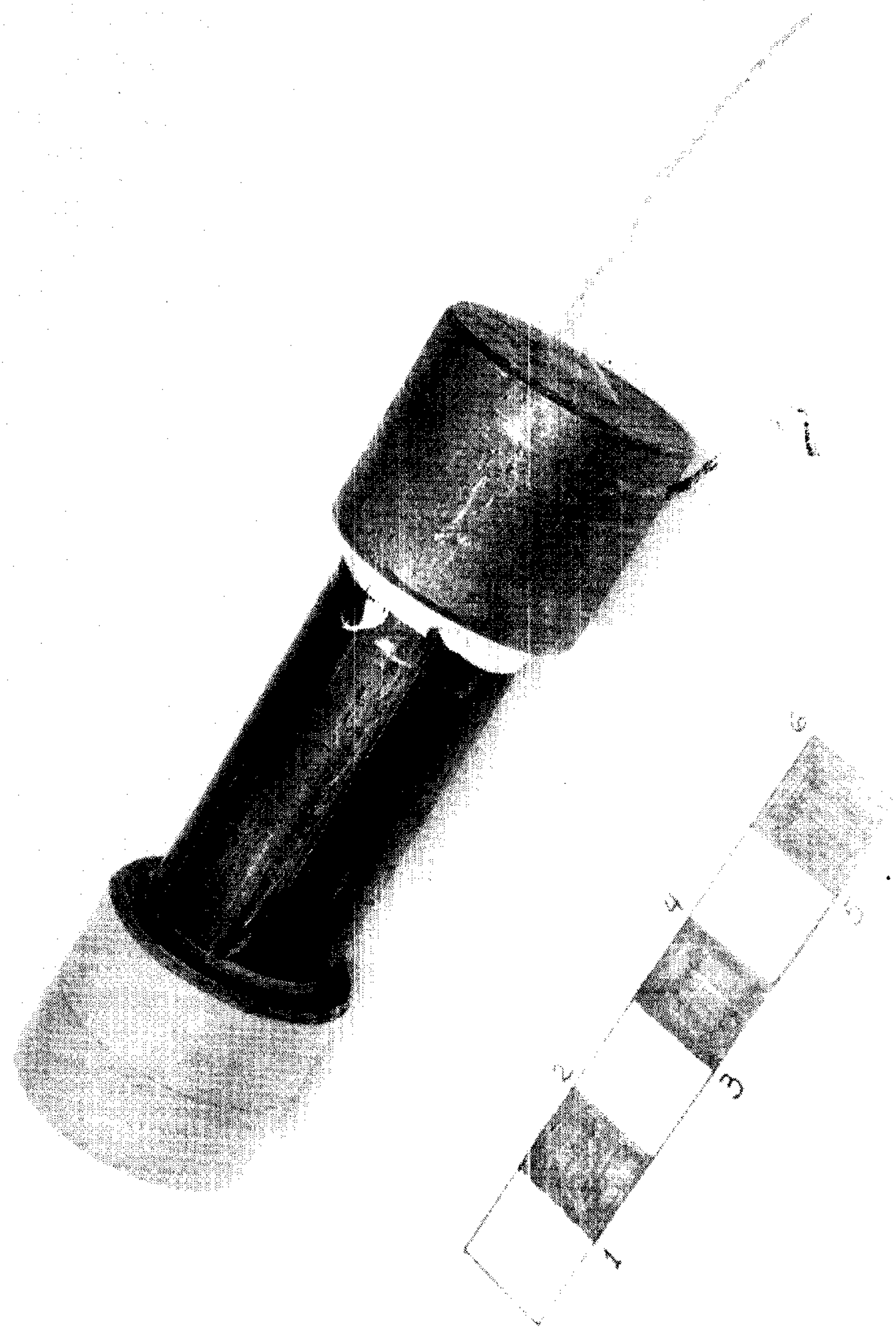


Figure 4-52. Typical HE Equivalency Test Container

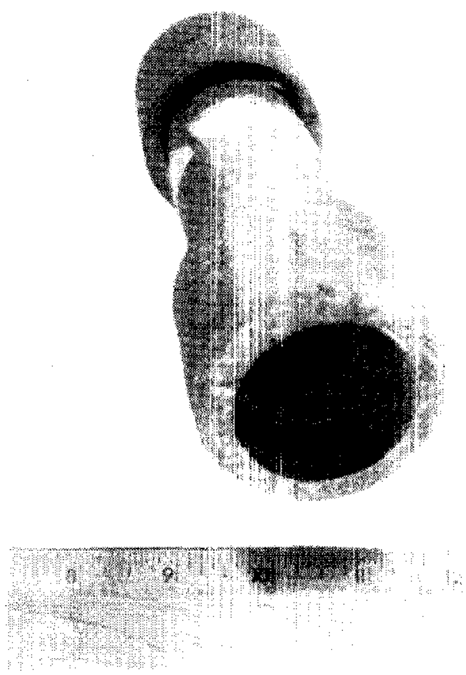


Figure 4-53. C4 and Flaked TNT 50 gram Sample Typical Results

Figure 4-54. 75 gram Sample C4 and Flaked TNT Typical Results

Figure 4-55. Typical Fragmentation of 100 gram Samples of C4 and Flaked TNT

4.4.6.3 Comparison of Fragments of High Explosives Versus Pyrotechnics

In this series of tests, the pyrotechnic composition mass was maintained at 100 grams. The high explosive samples were varied in weight (50, 75, 100 grams) in an attempt to duplicate the fragmentation characteristics of the pyrotechnic sample.

The visible fragmentation distribution of typical 100 gram pyrotechnic samples and 100 and 50-gram samples of HE is included in Figures 4-56 and 4-57. It is obvious that the fragmentation patterns of the 100 gram sample of HE bear no resemblance to that of pyrotechnic compositions. The most severe pyrotechnic results are similar to that observed with 75-gram samples of HE and the more typical pyrotechnic results resemble the fragmentation characteristics of 50-gram HE samples.

4.4.6.4 Vessel Rupture Strength Effects

The effects of vessel rupture strength on the fragmentation characteristics was studied for pyrotechnic and HE samples. The rupture strength was adjusted by varying wall thickness as shown in Figure 4-58. The length to diameter ratio is relatively constant in the various configurations and the material characteristics are identical. The tests were conducted with constant sample mass (100 grams), thus only the wall thickness distinguished the various tests.

The relative results were similar for HE and pyrotechnics. The thin walled vessels fractured into the greatest number of fragments and, as can then be predicted, the smallest mass fragments. Conversely, the thick walled vessels tended to maintain their integrity.

4.4.6.5 Fragmentation Velocities

Fragmentation velocities for pyrotechnics and high explosives were measured by time of flight techniques. The method used was a simple switching device and a discrete count step switch method. A start signal is generated when a wire circumventing the standard HE equivalency vessel is ruptured causing an open circuit. Fragments within a certain solid angle impinge on a sensor panel situated a known distance from the source (Figure 4-59). Impact on a panel (one of eight) produces a closed circuit condition which is also recorded. A before and after photograph of the impinged panels is shown in Figure 4-60. A simple circuit (Figure 4-61) permits recording all signals on a single recording channel by distinguishing the signals via pulse amplitude. Typical recorded signals are shown in Figure 4-62. The difference in the time between start and impingement signals divided into the distance from source to panel provides a measure of the average velocity of the fragment when traversing this distance. The results of these tests revealed as

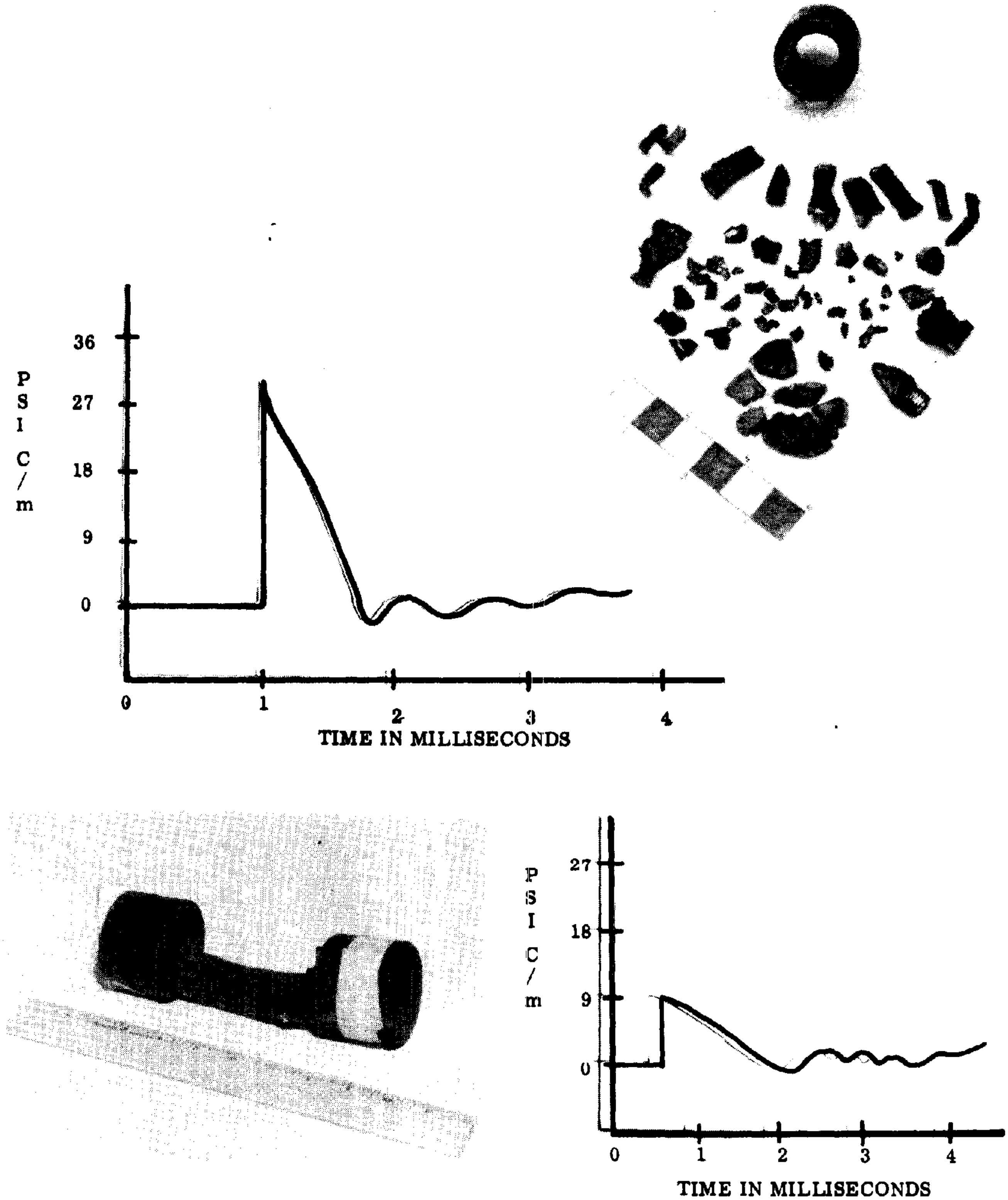
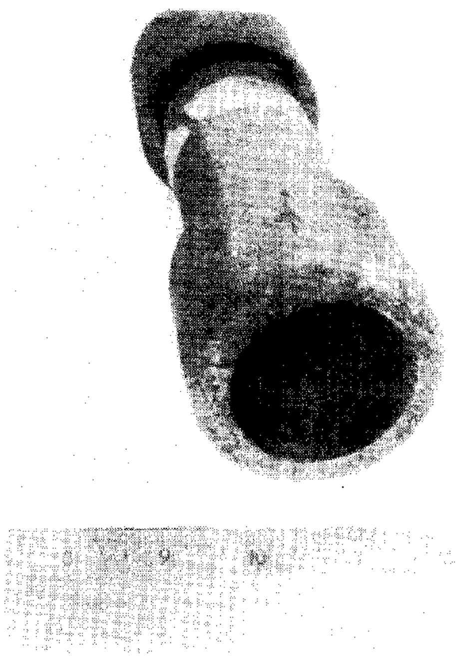
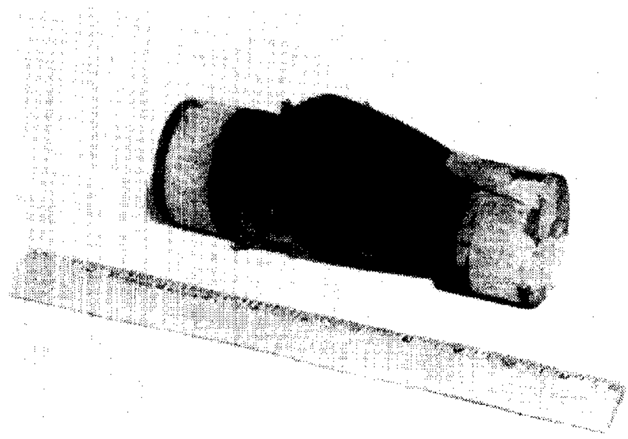


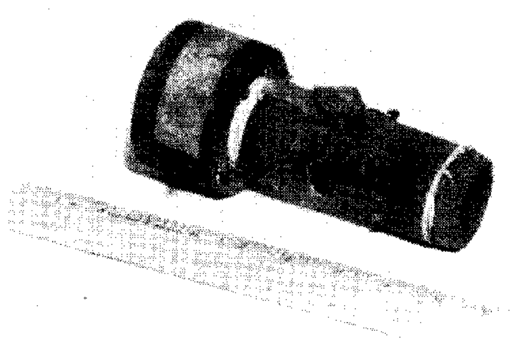
Figure 4-56. Typical Fragmentation Characteristic and Blast Pressure Profiles of 100 gram Samples of HE and Pyrotechnics



Example of Effect of HE 50 gram Sample



Example of Most Severe Pyrotechnic 100 gram Sample Effect



Example of Typical Pyrotechnic 100 gram Sample Effect

Figure 4-57. Typical Fragmentation from High Explosive and Pyrotechnic HE Equivalency Tests

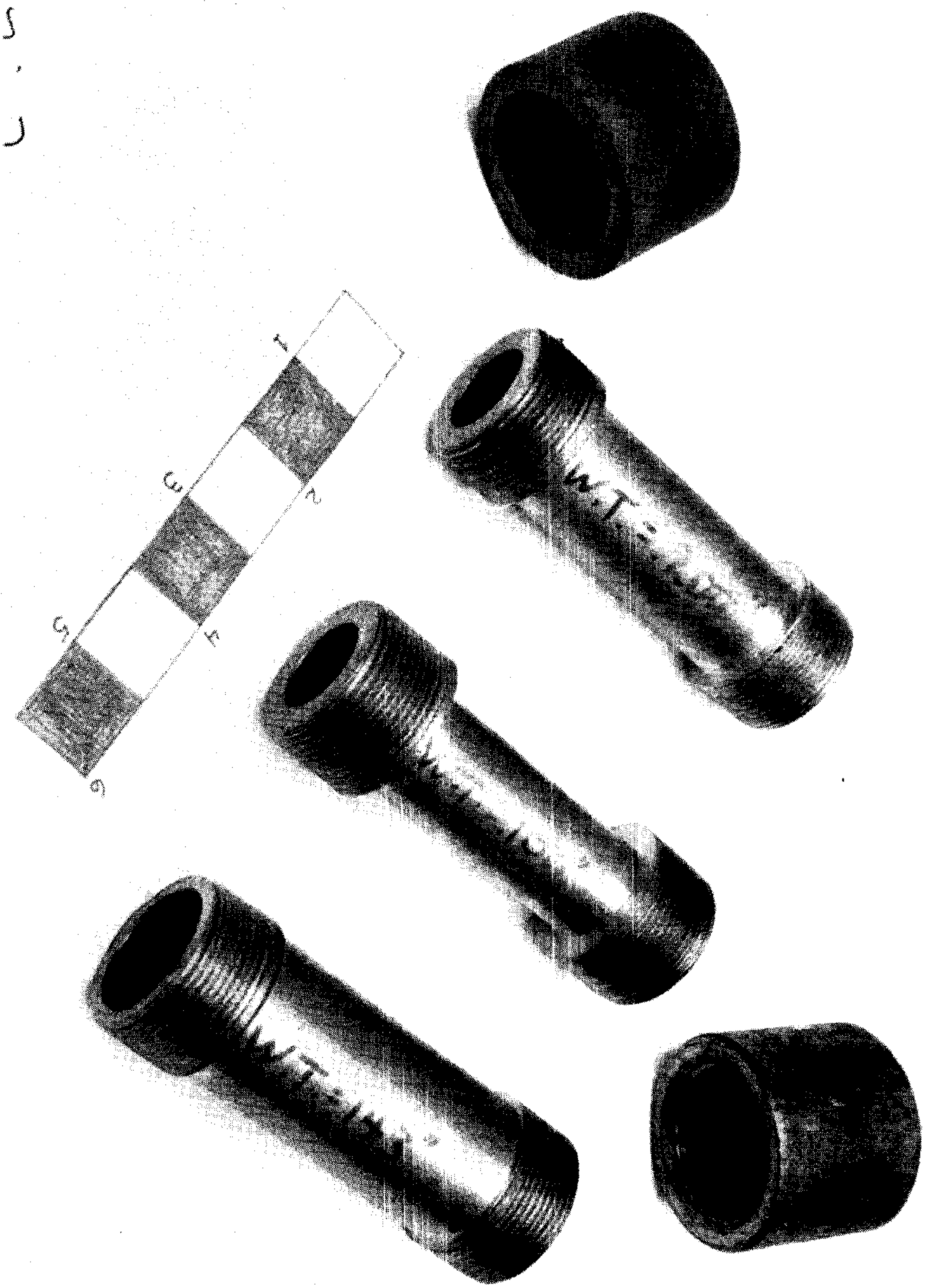


Figure 4-58. Vessel Configurations Used in Tests to Compare Effects of Variations in Ruptuse Strength

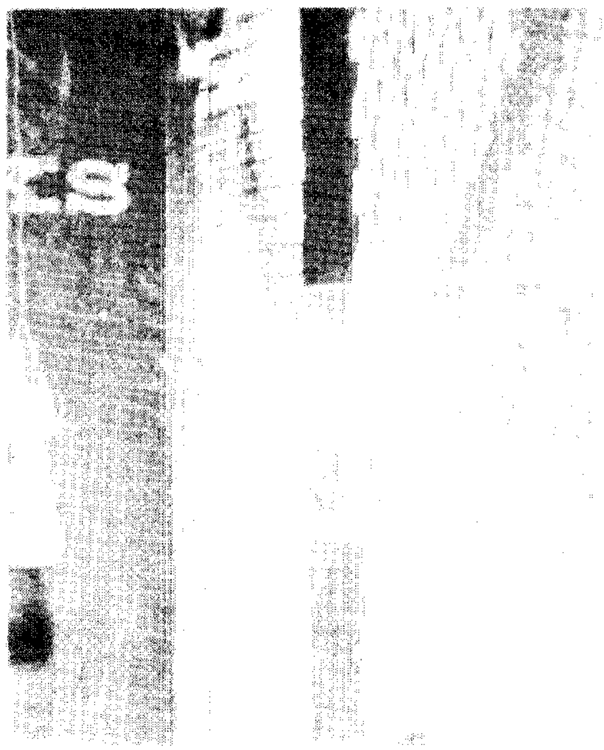


Figure 4-59. Discrete Fragment Method Single Fragment Measurement (Before)

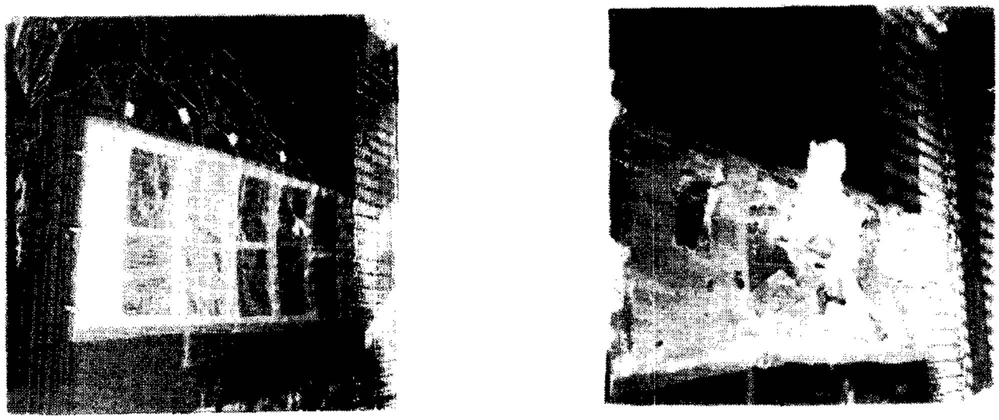


Figure 4-60. Discrete Switch Method Fragment Measurement (Before and After)

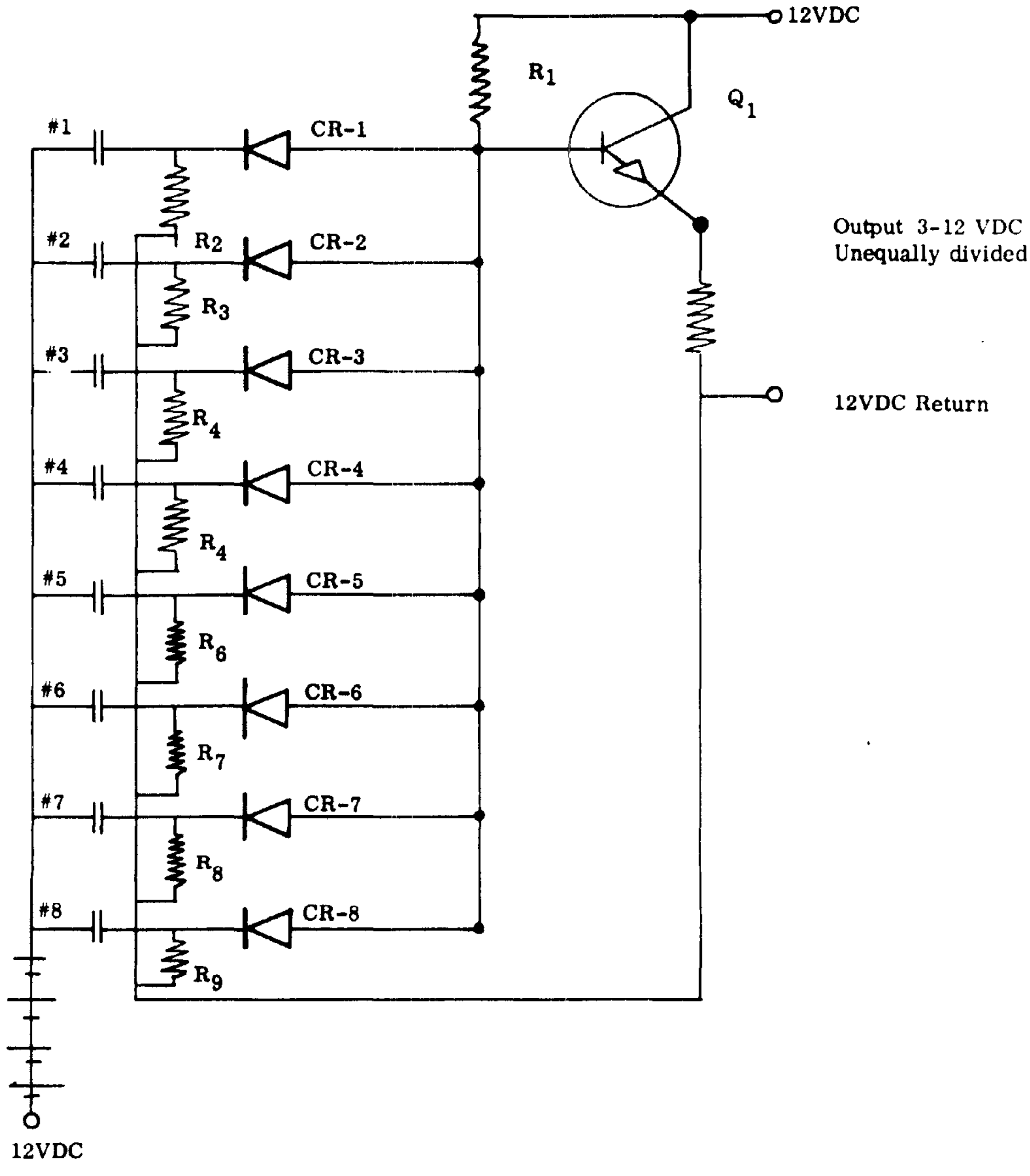


Figure 4-61. Discrete Switch Circuit

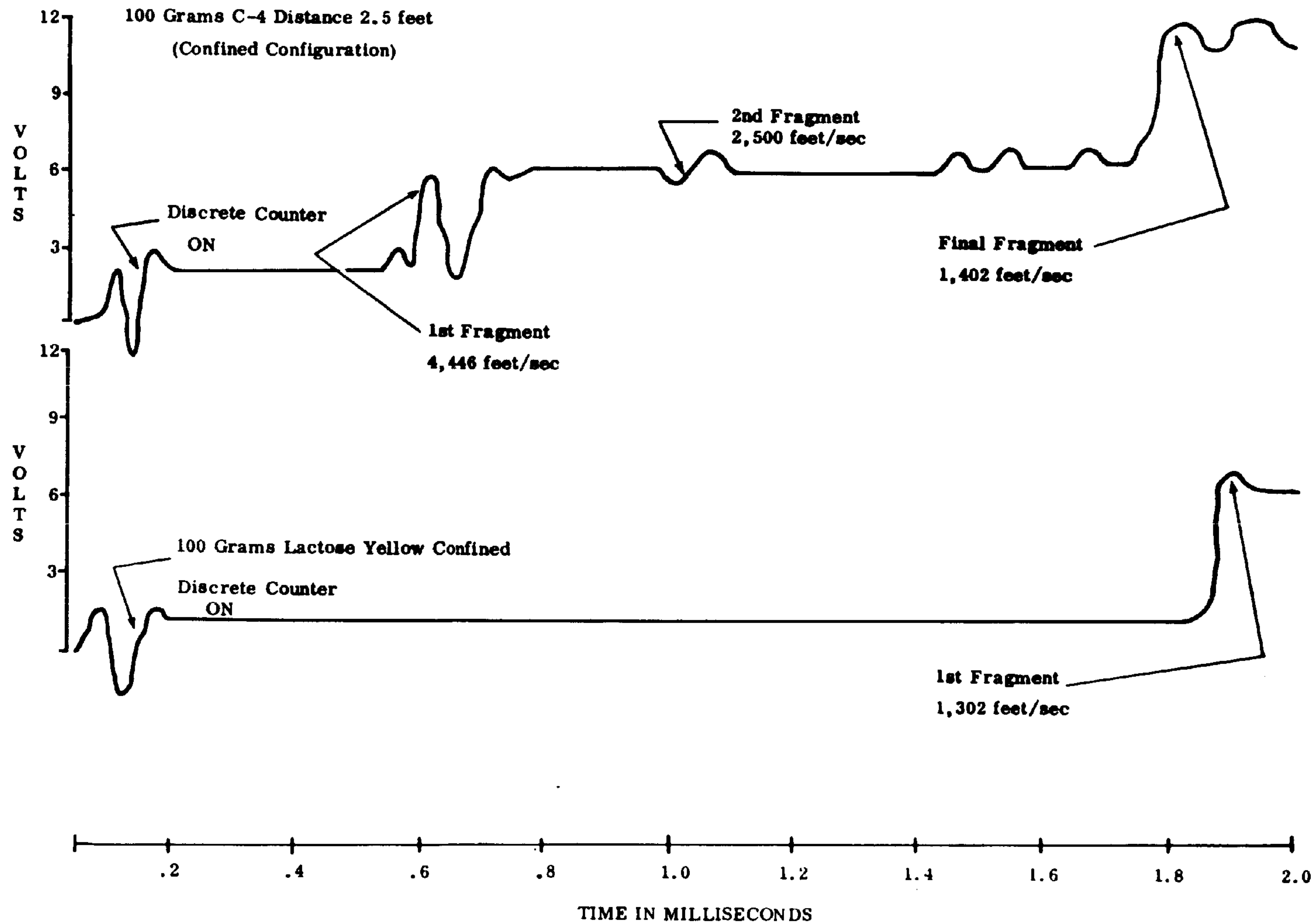


Figure 4-62. Comparison of Fragmentation Velocities - 100 grams Lactose Yellow vs 100 grams C-4

expected, that the velocity of the HE test fragments is much greater than that of the pyrotechnics samples; however, both types of fragments were in the supersonic range. Figure 4-63 shows the values obtained using the standard Celotex equation method for determining fragmentation velocity.

This series of comparisons indicated the following:

a. Pyrotechnics and High Explosives reacting in the same type of container have distinctively different fragmentation characteristics.

b. When the weight of high explosive in a given type of container is reduced as some function of the assumed HE equivalency of a given pyrotechnic the differences in fragmentation characteristics diminish.

In summation, tests conducted in these and other series have not indicated what is sometimes referred to as a "characteristic type" of fragment from High Explosives. They have indicated that the fragmentation characteristics more closely relate to the charge mass ratio, i.e., density and mass of charge versus density and mass of confining media. In this context, the less dense charge (pyrotechnic) develops an order of magnitude less pressure.

4.4.7 EFFECTS OF SAMPLE DENSITY ON TNT EQUIVALENCY

The information required to determine the depth of the material in the confinement vessel was recorded before capping. The depth multiplied by the cross sectional area (a constant for all vessels) would then establish the volume occupied by the material, which when divided into the mass of material would establish its density. The variation of the depth measurements was about 1/16 inch about a mean value for a given mass of a given pyrotechnic which is also the estimated measuring error. Therefore, no variations in density were detected. After capping the vessel and transporting and handling it, the material density may have been affected by a self-tamping action of the upper material on that below it; thus, the deeper samples may have a different average density than the shallower ones. But again, experience has indicated that the effect is small; therefore, densities at test time can be considered constant for a given material.

Any attempt to establish density variation effects a TNT equivalency with a given material is thus thwarted by a lack of precision density data. Also, even if the variations in depth of 1/16 inch were significant, the net effect on density is 1 or 2 percent (a typical depth is 5 inches).

Density variations between the different materials were not observed to be directly correlated to the TNT equivalency, as shown in Figure 4-64.

4.5 SPARK IMPINGEMENT TESTS

4.5.1 RATIONALE

Operational surveys have identified four generic hazard operations in the manufacture of pyrotechnics: filling, pressing, mixing, and reaming. Review of accident reports associated with

HE

EQUIVALENCY FRAGMENT -- VELOCITY USING SIMPLIFIED EQUATION

FRAGMENT NUMBER	VELOCITY	PENETRATION	MASS	THETA
07-1-1	3329.5	2.50	27.78	83.00
07-1-2	1736.5	4.00	407.42	99.00
07-1-3	2181.8	3.00	37.04	107.00
07-1-4	606.9	2.50	665.29	113.00
07-1-5	674.0	3.50	1821.02	107.00
07-1-6	1445.0	8.50	216.05	.00
07-1-7	6184.6	8.50	333.34	93.00
07-1-8	6924.1	8.50	40.12	99.00

Figure 4-63. Computer Tab Run of Fragment Velocity Calculations Using Simplified Celotex Formula

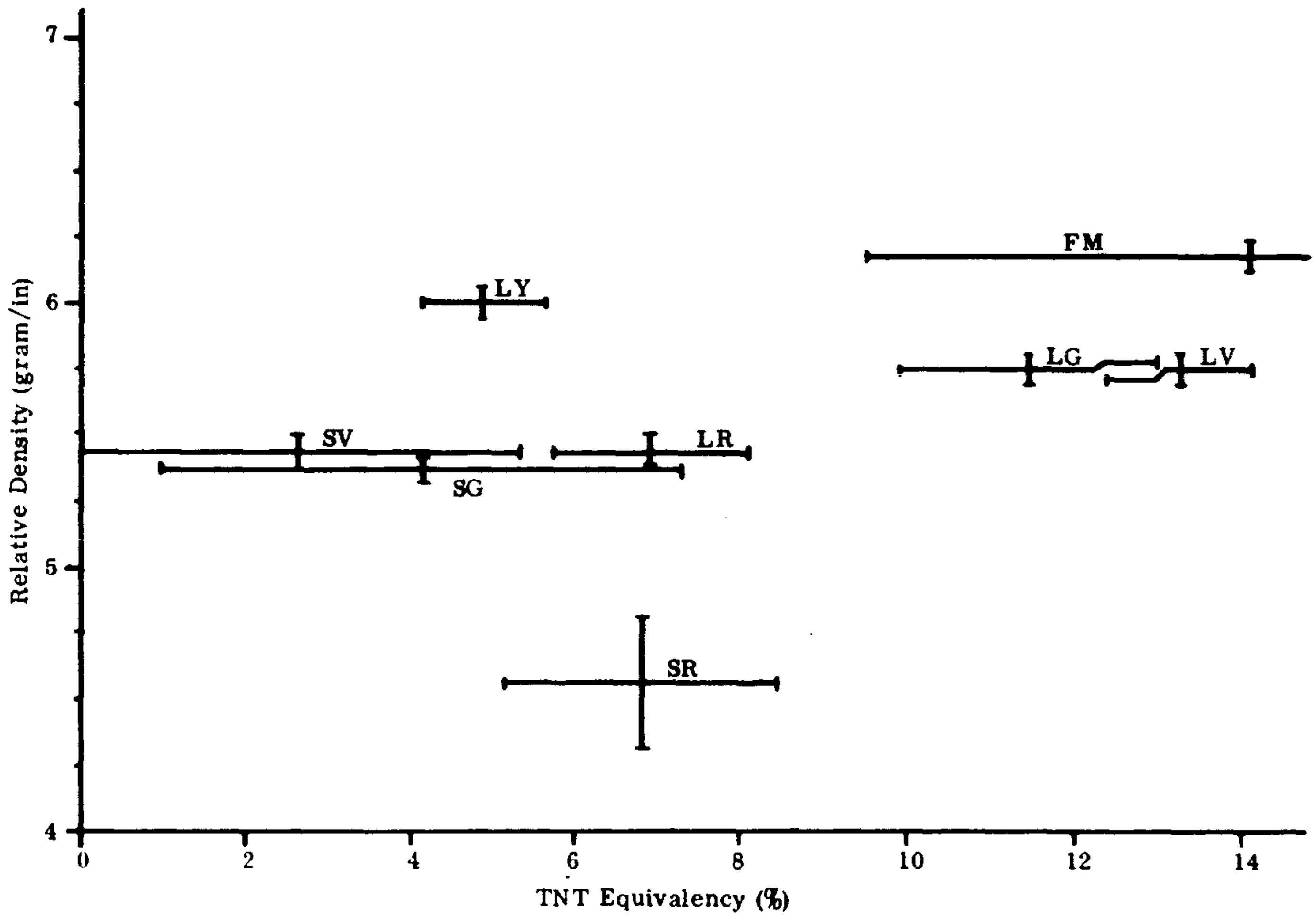


Figure 4-64. Density versus TNT Equivalency Value

these operations has shown that misalignment is a major common failure leading to ignition of the pyrotechnic material.

As a result of misalignment, friction between the misaligned components induces heating of the components through excessive rubbing and severe plastic deformation. As a result, sparks will be formed which may impinge upon pyrotechnic material during any of the normal filling, pressing, mixing, and reaming operations. If the sparks are sufficiently large and contain sufficient heat above a critical threshold level, the reaction will be self-sustaining and will ignite the material.

Tests were designed to investigate spark impingement by determining the relative sensitivity of various pyrotechnic formulations.

Through evaluations of spark impingement sensitivity of the various pyrotechnic formulations and investigation of spark suppression phenomenon, increased safety should result in the manufacture processes.

4.5.2 TECHNICAL APPROACH

The objective of this testing was to determine whether initiation of selected pyrotechnic materials could be induced by direct contact with frictional sparks. To accomplish this objective, frictional sparks were generated by applying a high carbon steel rod to a grinding wheel so as to direct sparks onto a cup containing a layer of pyrotechnic material. Ignition can then be detected by observation of a flame or excessive smoke.

The test materials were 10 gram batches of each of the following:

- Two Smoke Mixes - One sulfur base and one lactose base.
- Fuels - Sulfur, sugar, acetone, and heptane.
- Slurry 50-50 percent - Smoke mix plus heptane/acetone.
- Damp Mixture - Mixture of 10 grams of smoke mix plus 1 cc of water.
- Contaminated - Mixture of smoke mix plus 1 cc of light weight oil.

All powders were dried in an oven for 24 hours at 167°F. The damp and contaminated mixtures were formed after drying. Each material was tested four times; each time the distance traveled by the spark and the spark density was measured.

4.5.3 TEST RESULTS

The test results are as follows:

- None of the samples ignited.
- The distance of travel of the hot chips varied from 2 to 12 inches.

- The spark density varied in direct proportion to the degree of friction exerted by the carbon rod and the grind wheel.
- There was some detectable discoloration of the sample material in contact with the spark in most of the tests.

4.5.4 CONCLUSIONS AND RECOMMENDATIONS

Hot chips and sparks (produced using the techniques in this report) contain insufficient heat to ignite any of the materials tested. Local reactions were induced, but the "hot spots" formed were subcritical, thereby failing to be self-sustaining. Additional testing is recommended to ascertain the frictional spark intensities required to ignite various pyrotechnic compositions, solvents, solvent vapors, and solvent-pyrotechnic mixtures.

4.6 INSTRUMENTED PARR BOMB

A quantitative approach to establishing the detonability of the pyrotechnic through a study of the deflagration to detonation transition was attempted wherein the burning rate as a function of pressure was determined using the heat of combustion closed bomb (Parr Bomb).

The instrumentation technique utilized a Whittaker pressure transducer, a Tektronix 545 oscilloscope with Polaroid attachment and a hot wire ignition source in the Parr Bomb apparatus. Initiation of the hot wire ignition source and triggering of the oscilloscope were performed manually. The test setup is shown in Figure 4-65.

Measurement of the resultant pressures produced by the hot wire ignition of 1.0 gram of the loose granular pyrotechnic material using the modified Parr Bomb as the pressure vessel resulted in typical time-pressure profiles as shown in Figures 4-66 and 4-67. In each case the sweep time of the oscilloscope was set for 0.5 seconds per centimeter and the pressure at 100 psi per centimeter.

Test results were tabulated by material versus pressure, rise time and assumed burn rate from a linear interpolation of the rise time. Results are shown in Table 4-7.

A comparison of the closed bomb burn rate data with that taken from the tests performed in the HE Equivalency test series (paragraph 4.4) for equivalent sample materials is shown in Table 4-8. Since the HE Equivalency value is based on the time from initiation to rupture of the vessel and the Parr Bomb value on the peak pressure versus time, there is little or no direct comparison, except in relative ranking, which is compared with other sensitivity data in Table 5-1.

The small number of test values preclude ranking the Parr Bomb Pressure Rise with respect to hazard potential; however there is an indication that the lactose base compositions exhibit higher pressure in the Parr Bomb.

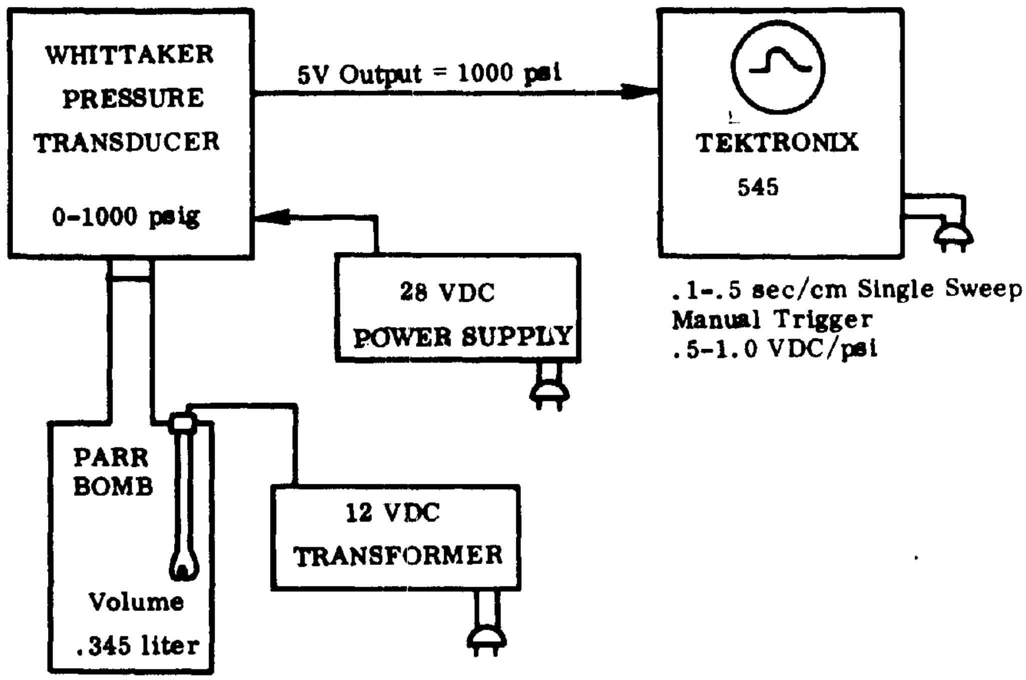
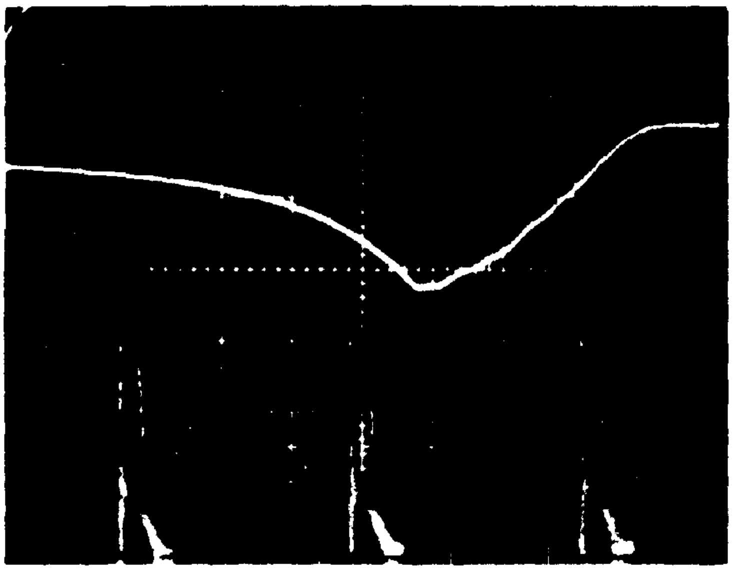


Figure 4-65. Pressure/Time Instrumentation

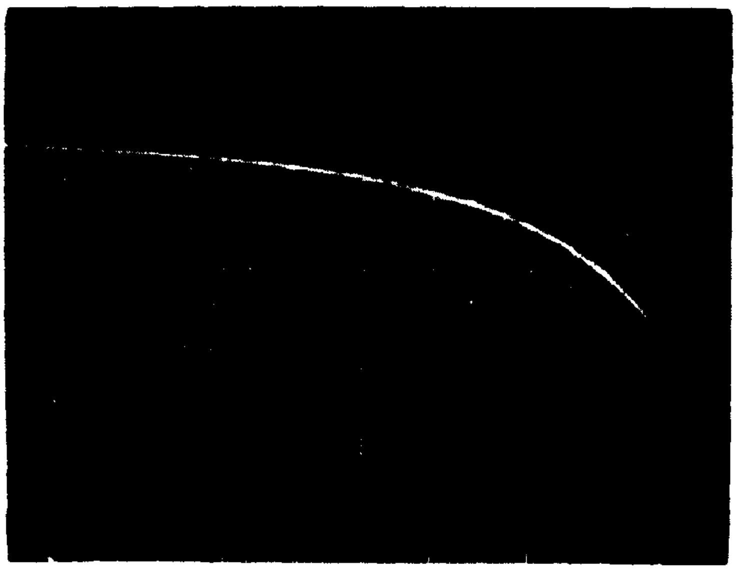
100 psi
per cm



0.5 sec/cm

Figure 4-66 Black Powder Pressure Trace psi/sec

100 psi
per cm



0.5 sec/cm

Figure 4-67 Lactose Yellow Pressure Trace psi/sec

Table 4-7. Parr Bomb Pressure Data

MATERIAL	PRESSURE PSIG	RISE TIME SEC	BURN RATE SEC/GRAM
Black Powder	280	.01	.01
$KClO_3$ S	240	.04	.04
Lactose Red	335	0.5	0.5
Lactose Green	340	0.7	0.7
Lactose Yellow	227	1.5	1.5
Lactose Violet	250	0.8	0.8
Sulfur Red	200	0.8	0.8
Sulfur Green	220	0.8	0.8
Sulfur Yellow	130	0.8	0.8
Sulfur Violet	200	0.9	0.9

Table 4-8. Parr Bomb Versus HE Equivalency

COMPOSITION	COOK-OFF TIME HE EQUIV. TESTS MILLISECOND	PARR BOMB BURN RATE SECOND	DATA PRESSURE (PSIG)
Sulfur Red	7.2	0.8	200
Sulfur Green	12.9	0.8	220
Sulfur Violet	14.6	0.9	200
Lactose Red	3.7	0.5	335
Lactose Green	34.1	0.7	340
Lactose Violet	21.0	0.8	250

Comparison "burn rates" from various tests and published sources are presented in Table 4-9. The function time value was extracted from the pyrotechnic-confined data collected during the detonation of high explosives discussed in paragraph 4.4. The Parr Bomb value was taken from data contained in paragraph 4.6 and the detonation rate was taken from Reference 7 "Explosives Series Properties of Explosives of Military Interest."

Table 4-9. Burn Rate Data Comparison

MATERIAL	(HE) FUNCTION TIME	BURN RATE PARR BOMB	DETONATION RATE
Black Powder	Not tested	100 gram/sec	400 meters/sec
TNT Flaked	.17 millisec	Not tested	6825 meters sec
Composition C-4	.12 millisec	Not tested	8040 meters/sec
Pyrotechnic (avg)	15.6 millisec	1 gram/sec	Data not available

Analysis of the values shows that the average confined burn rate for the pyrotechnics is in the neighborhood of one-hundred (100 times slower relative to the black powder and high explosives (TNT and C-4 confined).

Detonation rate data was not determined from the HE equivalency test series; however, in looking at the data in Table 4-9, it can be postulated that pyrotechnic compositions would exhibit a subsonic detonation rate.

SECTION 5
SEGMENT 4 - DATA REQUIRED FOR TESTING, EVALUATION, AND CLASSIFICATION OF PYROTECHNICS

5.1 INTRODUCTION

This segment of the program was structured to provide for correlation of the acquired data and information with respect to modification of a specification for evaluation and classification of pyrotechnic materials. Recognizing that the tests performed in previous phases and segments of the program are directly applicable to the final recommendations for specification modifications this segment has been divided into two parts:

- Data Comparison
- Conclusions and Recommendations

5.2 DATA COMPARISON

To better understand the potential hazards associated with the pyrotechnics an analysis was made of each test and its relationship with other tests performed. From this analysis an indication of a hazard index will provide the basis for criteria to be applied to pyrotechnics. An eventual military specification prepared exclusively for the evaluation and classification of pyrotechnics would be advisable. A matrix of the tests performed, their individual hazard ranking and the material tested is shown in Table 5-1.

Each test is individually ranked numerically from the least to the most hazardous value. The following discussions of each test and comparison with other tests are based on the comparison of values only.

5.2.1 DIFFERENTIAL THERMAL ANALYSIS (DTA)

This test examines the ignition temperature of the sample material through the application of controlled heat rise to the temperature at which decomposition occurs. Since the greatest hazard with respect to ignition is relatable to the lowest temperature the ranking reflects Lactose Green as the least hazardous and HC White Smoke and Fuel Mix as the most hazardous.

5.2.2 HEAT OF COMBUSTION (PARR BOMB)

This test was utilized to examine the quantity of heat in calories liberated by the combustion of 1 gram of the sample material. Ranking is based on the greater the value of heat liberated the greater the hazard. By this method, it is shown that HC White Smoke and Fuel Mix are the least hazardous material and Lactose Red the greatest. The heat of combustion for TNT has been determined to be 3620 calories per gram and would therefore, by comparison with pyrotechnic materials, be more hazardous. It does not necessarily apply that there is a direct relationship between the TNT and pyrotechnic since the rate of heat liberation could

Table 5-1. Sensitivity Ranking by Various Test Methods

SAMPLE MATERIAL	MILIT. CLASS.	DTA °C		STD. PARR CAL/GRAM		INSTR'D PARR PRESSURE (psi)		IMPACT (ØI) 10" DROP		ELECTRO-STATICS JOULES		TNT (C-4) %		TOTAL VALUE
		DATA	RANK	DATA	RANK	DATA	RANK	DATA*	RANK	DATA	RANK	DATA	RANK	RANK
Lactose Red	2	197	6	2988	10	335	7	0-1-9	3	.236	1	4.90	6	33
Lactose Yellow	7	217	3	2763	8	227	5	1-3-6	9	.102	9	3.58	5	39
Lactose Violet	7	210	4	2345	6	350	6	1-2-7	8	.200	2	9.44	9	36
Lactose Green	2	332	1	2960	9	340	8	0-1-9	4	.121	7	7.63	8	37
Sulfur Red	2	201	5	2282	4	200	2	0-6-4	7	.154	4	5.00	7	29
Sulfur Violet	7	221	2	2294	5	200	3	2-5-3	10	.161	3	1.74	3	26
Sulfur Green	2	196	7	2487	7	220	4	0-4-6	6	.131	5	2.72	4	33
Sulfur Yellow	2	196	8	2275	3	130	1	0-5-5	5	.113	8	**	1	26
HC White Smoke	2	193	9	939	1	-	-	0-0-10	1	.122	6	**	2	19
Fuel Mix	7	193	10	1000	2	-	-	0-0-10	2	.100***	10	11.44	10	34

1 - Least Hazardous

10 - Most Hazardous

* Explosion - Decomposition - No Reaction

** Failed to Ignite Under Test Conditions

*** Not Tested - Assumed Value

NOTE:

Ave. Value for Class 7 Compositions - 33.5

Ave. Value for Class 2 Compositions - 29.5

Ave. Value for Lactose Compositions - 36

Ave. Value for Sulfur Compositions - 28.5

vary from one to another. Values for the particular pyrotechnics in this program have not previously been determined although values for several binary systems and fuel oxidizer combinations are given in Engineering Design Handbook, Military Pyrotechnic Series, Part One, Theory and Application. (See bibliography)

Further research into modified applications of the Parr Bomb to determine pressure rise and burn rate characteristics of the pyrotechnic materials is recommended. Paragraph 5.2.3 discusses some of the work done on an instrumented Parr Bomb.

5.2.3 INSTRUMENTED PARR BOMB

The instrumented Parr Bomb test was discussed in Section 4.6. The data is summarized in Table 5-1 and shows the variation in pressure for the various lactose and sulfur base compositions. The most hazardous appears to be lactose green with a value of 340 psi and the least hazardous sulfur yellow with 130 psi. As stated previously a tremendous potential exists for a closed bomb type test for classification and evaluation determination. There is a possibility that a modified Parr Bomb instrumented with a sophisticated instrumentation/data acquisition system may be substituted for the HE equivalency test.

5.2.4 IMPACT SENSITIVITY (PHASE I)

Statistically the results taken from a 20 test drop sampling are inconclusive. The population (quantity) of tests should be increased to permit better statistical correlation. For the purpose of this comparison, only that data applicable to the 10 inch drop as taken from the Phase I report are being considered. Data recorded reflect Explosion, Decomposition and No Reaction. Thus, the ranking proceeds from Lactose Red which shows O-Explosions, 1-Decomposition and 9-No Reactions to Sulfur Violet with 2-Explosions, 5-Decompositions and 3-No Reactions.

5.2.5 ELECTROSTATICS

This test was performed to measure the sensitivity of the pyrotechnic materials to ignition by electrostatic charge. Sensitivity rankings begin at .236 Joules as the least sensitive and proceed to .102 Joules for the most sensitive. Lactose Red is the least hazardous and Lactose Yellow the most hazardous by this test.

5.2.6 HE EQUIVALENCY

This test as performed on the pyrotechnic materials established two (2) individual and distinct values for HE equivalency in percentages. Each of the two values was obtained from 100 grams of sample and normalized to an equal mass of reference material. The references used were:

- Trinitrotoluene (TNT) flaked - confined detonation, 100 gram weights
- Composition C-4 (C-4) - confined detonation, 100 gram weights

Examination by comparison of equivalent weights of both reference and sample at a selected "Z" of 5.25 ($Z = \frac{R}{W^{1/3}}$) against published values of pentolite at the same scaled distance indicated that the reference material detonation was most nearly equivalent to the published data when 100 grams of Composition C-4 with a density of approximately 1.60 gram/CM^3 were confined* in a vessel having an L/D ratio of 2.57:1. Comparison of the magnitude of the normalized peak overpressures indicated that Sulfur Violet has the lowest HE equivalency and Lactose Violet has the highest.

5.2.7 SUMMARY ANALYSIS

With few exceptions which may be accounted for either by the small number of tests performed or by the likelihood of significant sample component ratio differences due to small sample sizes (milligram) used in some tests, there is a fairly close correlation between the test results which measure the initiation sensitivity of the material. The results of the Differential Thermal Analysis, Impact, and Electrostatic tests show a close correlation in ranking of seven out of eight of the samples. Only Lactose Violet varied greatly in the relative rankings in the three tests.

In summary the reference data collected in this program establish the feasibility for use of small explosive charge diameters and configurations to be used for experimental purposes in determination of the hazard potentials of many compounds and materials. Our experience indicates that with utilization of proper instrumentation techniques and small charges the need for large scale pyrotechnic testing can be greatly reduced. The HE Equivalency values obtained in these tests are in the range from 0-12 percent. These low values characterize the relatively low efficiency of pyrotechnic materials for production of a blast wave.

5.3 CONCLUSIONS AND RECOMMENDATIONS

5.3.1 GENERAL

The objective of this section is to recommend modifications to current TB-700-2 methods and propose new tests and techniques in order to establish a definitive pyrotechnic hazards classification test program. This program is to be consistent with the particular properties inherent with pyrotechnic compositions and materials which characterize their potential for inducing a hazardous situation. The hazardous situations involving pyrotechnics are not limited to those within the TB-700-2 scope; those being transportation, handling (loading, unloading, and stacking), and storage.

* "In a cardboard tube"

5.3.2 TB 700-2 CRITERIA

Our analysis of the TB 700-2 classification techniques for these situations is that the existing document does provide reasonable classification procedures with appropriate simulation criteria for conditions associated with storage, transportation and handling. However, the information attained from these tests is insufficient to meet the needs of operating personnel in evaluating process station hazards. Additionally present methods do not involve sufficient instrumentation to provide the quantitative measurement of results desired. This inadequacy results in much latitude in interpretation of results and a general lack of quantitative classification criteria. The other primary objection to the current TB 700-2 approach is its failure to base its classification criteria to an appropriate phenomenological formation. The ICT sequence technique provides such an approach. Fortuitously the current test series was observed to adequately provide a measure of some of the elements of the ICT sequence. The appropriateness of the current interpretation criteria to establish the classification of materials by ICT parameters is questionable and requires reevaluation - particularly in the case of pyrotechnics. The current heuristic approach to pyrotechnic hazards classification should be changed. The data and results of this study should provide a significant fraction of the input required to effect this change.

As mentioned earlier the TB 700-2 applicability spectrum excludes many severe environmental situations such as occur during processing/manufacturing and development. The Phase II (GE-MTSD-R-058) portion of the current Hazards Evaluation Program provided a comprehensive analysis of the hazards involved during the manufacturing processes. One of the primary conclusions from that study was that establishment of material properties, particularly as related to the extreme environmental conditions of confinement, pressure, heat, friction, etc. encountered during manufacture, is required to establish appropriate safety criteria to optimize safety and cost effectiveness.

In addition the current TB 700-2 test program excludes determination or consideration of the potential for electrostatic discharge ignition of materials. This ignition mechanism has been studied in detail in an electrostatic vulnerability program. E8 and XM15/XM165 clusters were the items evaluated (GE-MTSD-R-052 and 057), but there is general applicability of the techniques and tests used in that study. The reader is referred to the aforementioned reports for the conclusions and recommendations resulting from those studies.

5.3.3 RECOMMENDATIONS FOR MODIFICATION OF EXISTING TESTS

5.3.3.1 Impact Sensitivity Test

Specifically we recommend that the impact sensitivity apparatus be modified to include a strain guage positioned to measure the expansion of the confining cup during impact. Our studies indicate that it should be possible to descriminate between an explosive and a non-explosive by this technique, thus providing a positive criteria on which to base the order of response. Once thoroughly investigated and proofed, this reading should replace the current sonic characteristic criteria for interpretation of results.

The quantity of material used in this test should be reevaluated to consider the statistical fluctuation in relative concentrations of the components of a small composition sample. For example, if one considers a compound consisting of components which are composed of identical grain size distributions and if the initial batch from which the sample is taken is homogeneously mixed and of exactly the correct proportions, then the sample size must include enough gains n so that the permissible error in proportions is maintained. Statistically this requirement results in the condition that

$$\% \text{ error} \leq \frac{100}{\sqrt{n}}$$

Thus if a 1 percent error is specified at least 10,000 grains must be included in the sample. If the sample is prepared by passing through a #200 sieve (0.003 inches opening) and assuming the average grain volume is 2×10^{-7} cm^3 with an average density of 2 gm/cm^3, then a minimum sample mass of 4 mgs is required for 1% errors. This example is very idealized (for example there is no requirement in TB 700-2 as to sieving) and a realistic application of this analysis will deviate significantly from the assumptions made, thus it is recommended that the sample size be increased to the 20 to 50 mgm range subject to further study and analysis.

5.3.3.2 Standard Detonation Test

As recommended previously some modification to the standard detonation test might improve interpretation of results. Specification of a standard type of lightweight container, and the utilization of a go-no go gage to measure minor deformation will help in providing for correlation of data. However this test is not recommended for the evaluation of granular pyrotechnics.

5.3.3.3 Ignition and Unconfined Burning Tests

This test, if retained, should be modified as previously suggested, to provide for a standardized (e.g. filter paper) container for the granular pyrotechnic materials. Some specification should be made to minimize variance in the properties of the fire to which the single or 4 block samples are subjected such as the use of alcohol/glass wool rather than kerosene soaked sawdust.

Again this test is not recommended for granular pyrotechnics, for the reasons listed, and because the mass variance from 1 to 4 blocks is not believed sufficient to be meaningful.

5.3.4 THERMAL STABILITY TEST

This test is useful in determining whether a material may ignite in the normal thermal extremes (excluding fire) of the transportation environment. The following modifications are recommended for its improvement.

Insertion of thermocouples in sample and in the oven to indicate temperature excursions, as discussed previously, will help in obtaining more useful information, as will the development of a weight loss specification as a "significant change of state."

It is believed however, that a DTA test is more quantitative and should replace the thermal stability test, particularly since other process studies should have indicated a potential "cook off" at these temperatures.

5.3.5 CARD GAP TESTS

The card gap test is of doubtful value in the classification of pyrotechnics. Some of the possible modifications as discussed previously, i.e., longer length of sample, softer witness plate, etc., will help improve its usefulness but it is not recommended for classification of pyrotechnics. More useful information may be obtained by using the modified HE equivalency test fixture utilized in this program.

5.3.6 END ITEM TESTS

End item tests are considered useful in evaluating storage and shipping hazards, and should be retained, including a requirement to measure overpressure and impulse, with an acceptable transducer system. Additionally, high speed movie film records should be made.

5.3.7 ADDITIONAL RECOMMENDED TESTS

5.3.7.1 HE Equivalency

HE equivalency testing has provided a technique for correlating the blast energy output of pyrotechnics to that of a standard HE (TNT and C-4 in our study). The confinement afforded by the vessel provides the capability to obtain high pressures during reaction which affect the reaction rate. Unfortunately the effect of the vessel on the shock wave produced is to extract some energy from it to fissure the walls and supply a contribution due to release of the ambient internal pressure. However, if the vessel is properly calibrated by High Explosives tests, the information obtained will be of value, particularly in assessing operational station hazards. The relevancy of this test is obvious during certain manufacturing processes, where various degrees of confinement are maintained. Thus this test, while requiring further study to fully establish a validity interpretation criteria, is recommended as a classification test applicable to pyrotechnics.

While it has been stated that any composition capable of an exothermic reaction can be made to detonate, the results of tests conducted as part of this program are not conclusive.

As touched upon previously, the investigations of the smoke mixes concerned have shown that "worst case" confinement had to be achieved before any severe reaction was observed. Although under these conditions a characteristic pressure time record is obtained, it has been theorized that the overpressure resulting from the TNT equivalency test explosion is simply a pneumatic rupture of the test vessel. The test vessel in this case is 1 7/8 inch OD seamless tubing with a 0.22 $\pm$ .02 inch wall thickness, 5 1/2 inches long. The calculated burst pressure of this pipe is 6600 $\pm$ 600 psi. Some work has been done in the area of TNT equivalency determination for bursting pressure vessels by R. E. Olson, Safety Specialist of the Martin Company, Denver, Colorado and others. Table 5-2 is a tabulation of data which shows the TNT equivalency value in terms of pounds of TNT per cubic foot of tank (pressure vessel) volume for varying tank pressures. Using the nominal value of 6600 psi as the tank pressure and interpolating between the appropriate energy equivalencies in Table 5-2, a value of 4.056 pounds TNT per cubic foot of tank volume is obtained. Using the volume of the TNT equivalency sample vessel, 8.9 cubic inches, a TNT equivalency value of .0209 pounds or 9.5 grams is obtained. When compared to the 100 gram charge weight used for pyrotechnics a TNT equivalency value of 9.5% results. This compares very favorably to the average TNT equivalency values obtained in the test program reported in Section 4, which ranged from 1.7% to 11.4% (based on 100 grams of C-4 confined). Assuming nominal variation in rupture characteristics and material specifications, an even closer correlation can be made.

5.3.7.2 Hartmann Apparatus Test

It has been concluded that the Hartmann Apparatus represents a significant testing method for a general parametric evaluation of dust suspensions. As shown in Table 4-4, the results of the minimum concentration rankings appear consistent with other tests in indicating the material ease of initiation. For pyrotechnics it has been suggested that a more positive, volumetric type of ignition source (i.e. gun cotton) as opposed to hot wire, single spark, etc., is warranted. Additionally, selection of a larger size dust chamber would make the data less dependent of the characteristics of the dispersion system.

5.3.7.3 Differential Thermal Analysis (DTA)

The DTA test is believed to be a useful supplement to pyrotechnic classification tests. It provides for relative ranking of pyrotechnics to each other, to explosives, and to other less reactive hazardous materials. As shown in the tabulations of test data (Table 5-1), the results of such rankings appear consistent with other tests in indicating the materials ease of initiation (I). It is recommended as a criteria for hazards evaluation purposes.

5.3.7.4 Parr Bomb

This test both in the normal configuration and instrumentation has been discussed in some detail above. As previously pointed out above, the potential for this test as a candidate sensitivity/classification and evaluation test is probably the highest than for any other test.

Table 5-2. Energy Equivalent Table

Tank Pressure (psig)	Energy Equivalent in Pounds of TNT per Cubic Foot of Tank Volume
10	0.001238
20	0.002711
30	0.004591
40	0.00748
50	0.00936
60	0.01277
70	0.01458
80	0.01729
90	0.0203
100	0.0230
200	0.0566
300	0.09418
400	0.1340
500	0.1787
600	0.2252
700	0.2719
800	0.3211
900	0.3710
1,000	0.4150
2,000	0.4937
3,000	1.043
4,000	2.218
5,000	2.960
6,000	3.650
7,000	4.326
8,000	5.05
9,000	5.79
10,000	7.53
20,000	15.29
30,000	22.53

Note: To obtain the pressure vessel energy equivalent, multiply the energy equivalent per cubic foot by the vessel volume in cubic feet.

5.3.7.5 Summary

It is believed that the recommendations herein will provide an effective interim modification to pyrotechnic classification criteria. As further evaluation and testing proceeds, the basic ICT parameters referred to herein will be developed and quantified.